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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT NO. 14
For Quarter Ending October 15, 1968

prepared by
R. W. Harrison

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-6474
Robert L. Davies, Project Manager
Materials Section

NUCLEAR SYSTEMS PROGRAMS
SPACE SYSTEMS
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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prepared by
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approved by
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NUCLEAR SYSTEMS PROGRAMS
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Period July 15 to October 15, 1968
March 20, 1969

CONTRACT NAS 3-6474

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FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. R. L. Davies of NASA - Lewis Research Center is the NASA Technical Manager.

The program is being administered for the General Electric Company by E. E. Hoffman and R. W. Harrison is acting as the Program Manager. J. Holowach, the Project Engineer, is responsible for the loop design, facilities procurement, and test operations. Personnel making major contributions to the program during the current reporting period include:

Alkali Metal Purification and Handling - Dr. R. B. Hand, L. E. Dotson, and H. Bradley.

Gas Analysis - W. L. Hasty, Jr.

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from July 15, 1968 to October 15, 1968. The primary task of this program is to fabricate, operate for 10,000 hours and evaluate a T-111 Rankine System Corrosion Test Loop. Materials for evaluation include the containment alloy, T-111 (Ta-8W-2Hf) and the turbine candidate materials Mo-TZC and Cb-132M which are located in the turbine simulator of the two-phase potassium circuit of the system. The loop design will be similar to the Cb-1Zr Rankine System Corrosion Test Loop; a two-phase, forced convection, potassium corrosion test loop which has been tested under Contract NAS 3-2547.(1) Lithium will be heated to 2250°F by direct resistance in a primary loop. Heat rejection for condensation in the secondary potassium loop will be accomplished by radiation in a high vacuum environment to the water cooled chamber. The compatibility of the selected materials will be evaluated at conditions representative of space electric power system operating conditions, namely:

a. Boiling temperature, 2050°F
b. Superheat temperature, 2150°F
c. Condensing temperature, 1400°F
d. Subcooling temperature, 1000°F
e. Mass flow rate, 40 lb/hr
f. Boiler exit vapor velocity, 50 ft/sec
g. Average heat flux in plug (0-18 inches), 240,000 BTU/hr ft^2
h. Average heat flux in boiler (0-250 inches), 23,000 BTU/hr ft^2

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(1) Hoffman, E. E. & Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, Potassium Corrosion Test Loop Development Topical Report No. 67, R66SD3016, General Electric Company, Cincinnati, Ohio, May 1, 1968.

In addition to the primary program task cited above the program also includes capsule testing to evaluate advanced tantalum alloys of the ASTAR 811 type (Ta-8W-1Re-1Hf) in both potassium and lithium.

Also included in the program is the fabrication, 5000-hour operation and evaluation of a 2600°F, high flow velocity, pumped lithium loop designed to evaluate the compatibility of the ASTAR 811 type alloys, T-111, T-222, and the tungsten alloy, W-25Re-30Mo.

II. SUMMARY

Evaluation of the boiler material documented it for re-use and the boiler was subsequently repaired and postweld annealed.

A special welding chamber was installed on the T-111 Corrosion Loop facility and qualified for welding.

The repaired boiler was reinstalled in the loop and postweld annealed.

The loop is ready for reinstrumentation.

Testing of three ASTAR alloy lithium thermal convection capsules was initiated.

III. PROGRAM STATUS

A. T-111 RANKINE SYSTEM CORROSION TEST LOOP

1. Metallographic Examination of the Crack in the 0.375-inch Diameter T-111 Alloy Boiler Tube

The weld nugget section of the 0.375-inch tube butt weld containing the crack was removed from the boiler and ground longitudinally up to the crack area in preparation for metallographic examination.[(1)] The specimen was mounted in clear epoxy such that the tube could be examined longitudinally, transverse to the crack. The specimen was Automet* ground on 600 grit paper up to the fiducial marks which were placed on the specimen before mounting to indicate the location of the crack. Final polishing was accomplished using standard procedures.

The crack was observed to be intergranular and, as shown in Figure 1, View AA, did not extend completely from the OD to the ID of the weld nugget. The specimen was subsequently repolished removing only approximately 0.002-inches from the surface. The appearance of the crack was similar as shown in View BB of Figure 1 but shorter in depth. An additional polish was performed removing 0.006-inches of material from the surface. The crack appearance after this polish was considerably different and not as deep as observed previously as can be seen in View CC

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(1) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 13 for Period Ending July 15, 1968, NASA Contract NAS 3-6474.

*Automatic Metallographic Polishing Apparatus - Buehler Ltd., Evanston, Ill.

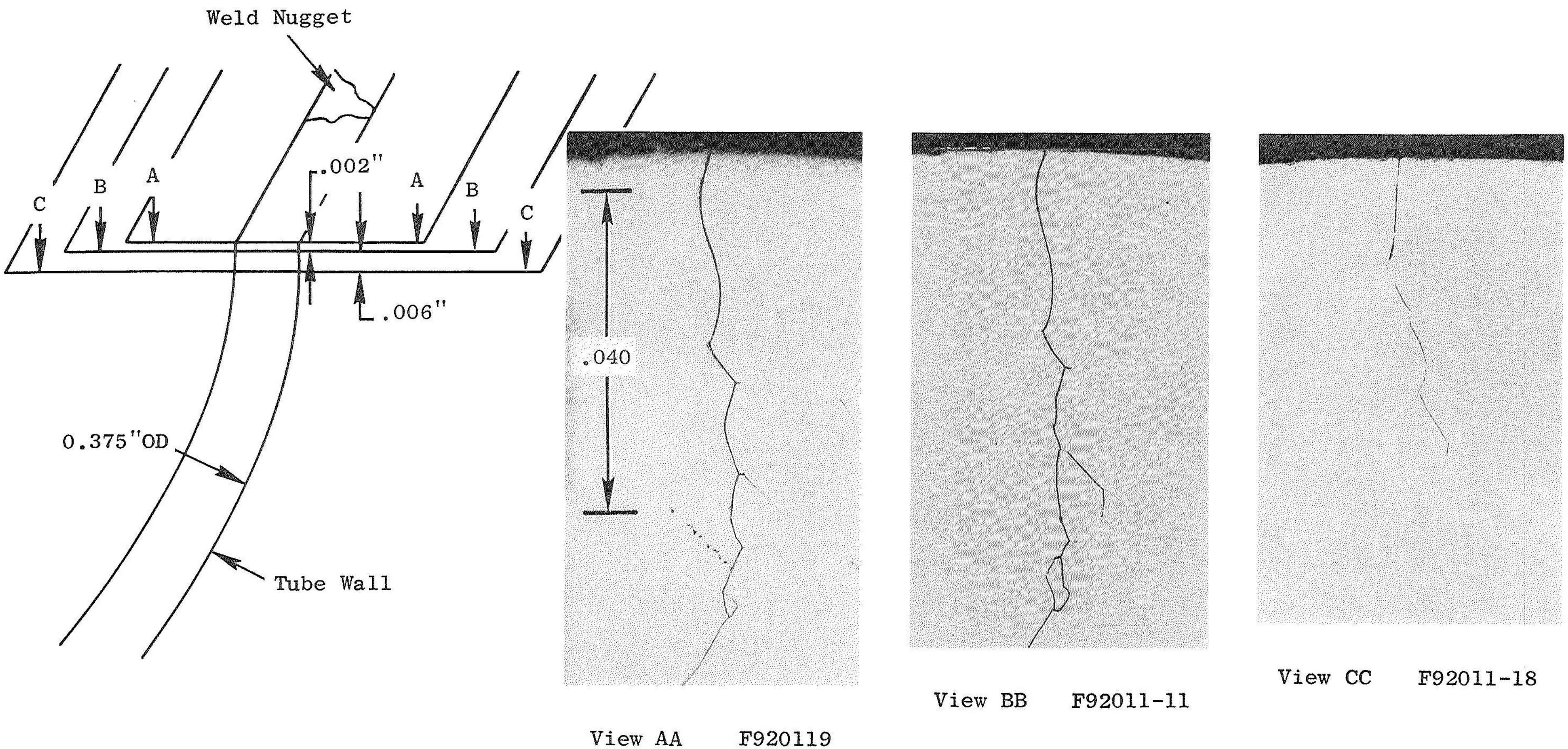

Figure 1. Intergranular Crack in the Weld Nugget of the 0.375-Inch Diameter T-111 Boiler Tube Removed From the T-111 Rankine System Corrosion Test Loop.

of Figure 1. It became clear at this point that the orientation of the specimen in the mount was such that the egress of the crack at the ID had been passed in the initial polishing step. In any event, examination of the crack at higher magnifications, performed between the polishing steps previously described, did sufficiently indicate the morphology of the crack.

The crack deviates from a simple shortest path route, and entire grains are delinated by the grain boundary separation, as can be seen in Figure 2. The appearance of grain boundary separation suggests some action of the lithium which has penetrated into the crack. The intermittent gaping of the grain boundaries, especially off from the main crack path and at the bottom of the crack as shown in Figure 2, is similar in appearance to lithium corrosion. Such a mechanism is difficult to understand since no corrosion by lithium has been observed in T-111 having such a low oxygen concentration (40 ppm). The role of lithium in the resulting metallographic structure is further exemplified in the microstructure at the bottom of the crack as shown in Figure 3. The grain boundary voids illustrated are difficult to explain in terms of cracking alone. The microstructures shown in Figures 2 and 3 are of areas in the specimen below the main crack path as indicated by their appearance as well as the fact the crack depth is decreasing with each additional polish.

The following conclusions have been derived from the metallographic examination:

(1) Although the cause of the crack is not evident, it is not believed to be initiated by alkali metal corrosion.

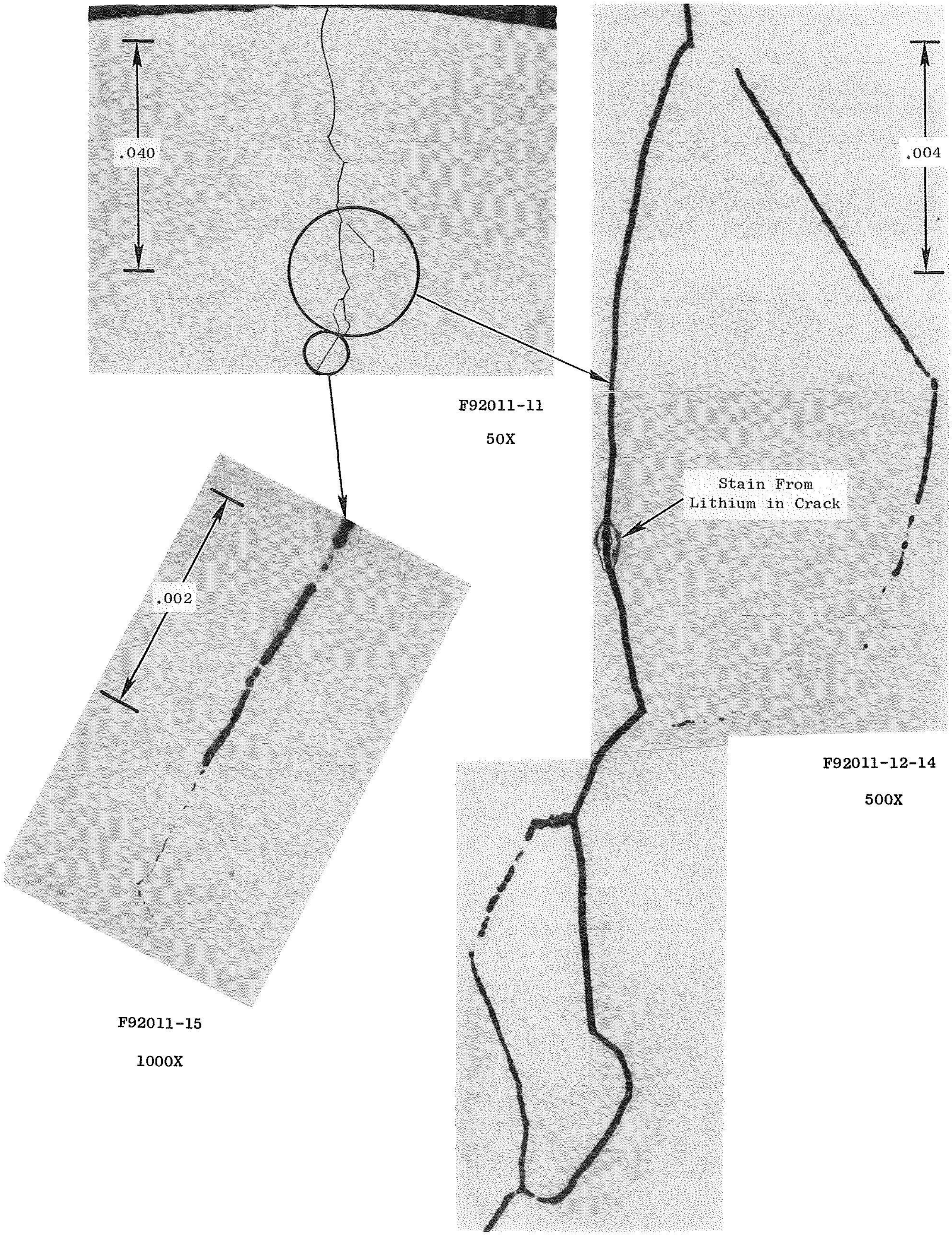

Figure 2. Intergranular Crack in the Weld Nugget of the 0.375-Inch Diameter T-111 Boiler Tube Removed From the T-111 Rankine System Corrosion Test Loop.

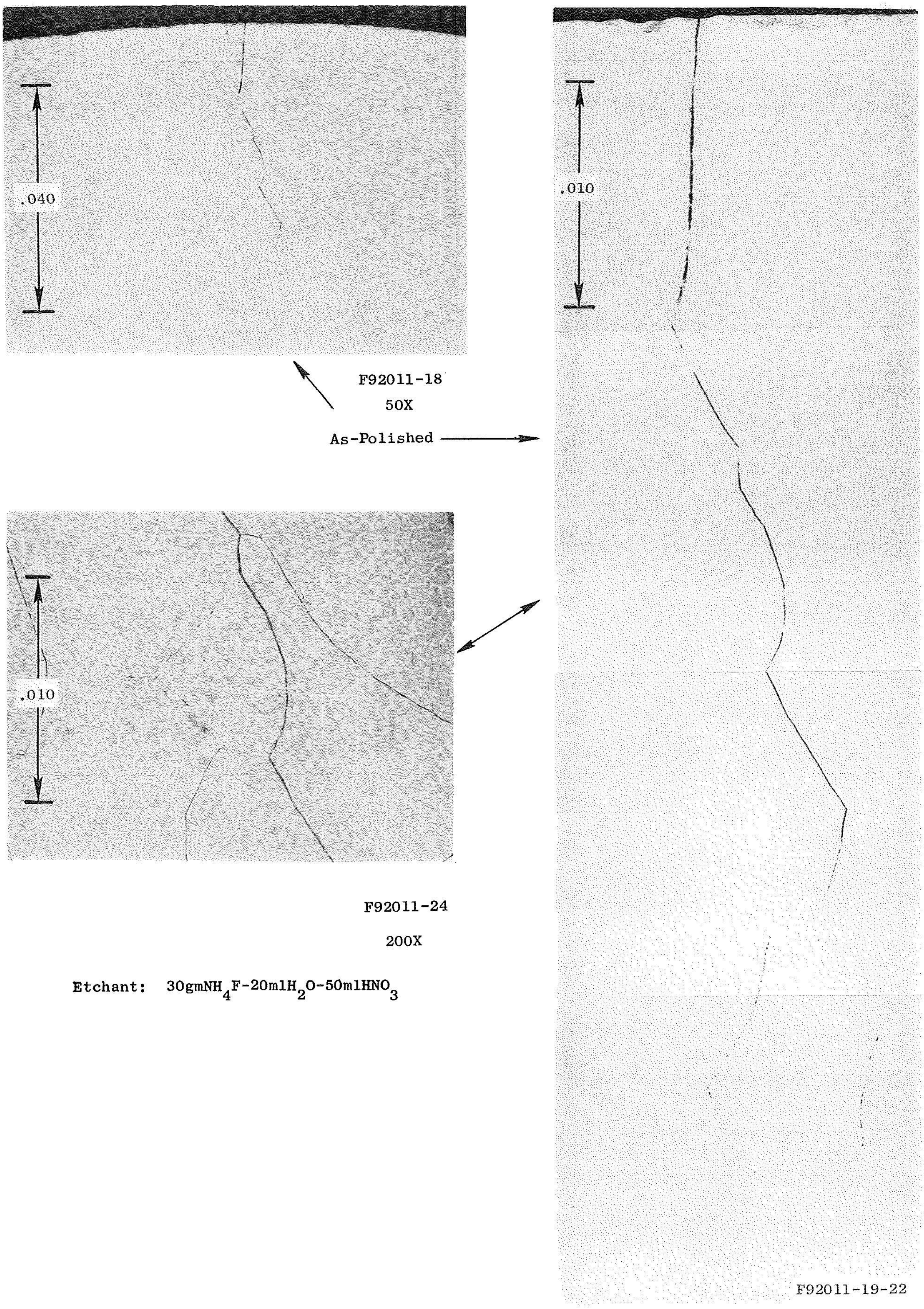

Figure 3. Intergranular Crack in the Weld Nugget of the 0.375-Inch Diameter T-111 Boiler Tube Removed From the T-111 Rankine System Corrosion Test Loop.

(2) The crack appears to have been initiated at the tube OD or lithium side and is completely intergranular.*

(3) The morphology of the area surrounding the crack appears to be influenced by the presence of lithium in the crack and if corrosion has occurred a mechanism is not understood.

Further examination of the crack in the weld nugget is planned using microprobe analysis and electron microscopy to determine possible compositional variations in the regions of the crack.

2. Evaluation of the T-111 Boiler for Possible Re-use in the Corrosion Loop

The location of the leak in the boiler tube was such that a suitable repair plan could be devised; however, evaluation of the boiler material was required to document it for re-use. Specimens were removed from the boiler in the vicinity of the crack for metallographic examination and spectrographic, interstitial, and microprobe analyses. The ductility of the T-111 was determined qualitatively by compression testing.

Metallographic examination indicated no changes in the microstructure of the T-111 specimens from the boiler as a result of the alkali metal exposure and ammonia cleaning. Sections of boiler tubing were flattened without evidence of cracking or change in ductility.

The interstitial analysis results obtained on specimens removed from the boiler are compared with that obtained on as-received tubing

*Recent tests have indicated that slight straining of T-111 weldments while hot (above 2500°F) results in grain boundary separation in the weld nugget and a small crack of this type may have been introduced during butt welding of the tube initially. A crack of this type could have propagated during bending of the tube in the fabrication of the boiler which was performed after welding.

in Table I. No major changes were noted as a result of the test exposure. Interstitial analysis of T-111 weld control specimens welded before and after the welding of the boiler, as part of NSP Specification 03-0025-00-A "Welding of Columbium, Tantalum and Their Alloys by the Inert Gas Tungsten Arc Process," indicated no contamination occurred during welding of the boiler.

Chemical analysis of the particulate matter found in the potassium drained from the loop indicated concentrations of Fe, Ni, Cr, Mn.[1] The possibility of contamination of T-111 loop materials by exposure to the particulate matter was checked by analysis for these elements. Microprobe traverses of the T-111 0.375-inch diameter boiler tube showed no traces ($\ll$ 1000 ppm) of Fe, Ni, Cr, or Mn. Subsequently specimens of 0.375-inch tubing were pickled, and the acid solutions analyzed spectrographically for Fe, Ni, and Cr. These results are given in Table II. The higher iron and nickel concentrations observed in the first analysis of the boiler material were not observed in the subsequent analysis. Approximately 0.001-inch thickness of material was removed by each pickling operation. The slight increase in iron and nickel concentrations in the first 0.001-inch of the boiler tube wall is not believed to be detrimental.

Based on the results of the various evaluations cited, repair of the boiler was recommended, and drawings of the modifications needed to re-use the boiler were prepared for NASA approval.

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(1) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 13 for Period Ending July 15, 1968, NASA Contract NAS 3-6474.

TABLE I.

INTERSTITIAL ANALYSIS OF 0.375-INCH T-111 TUBE SPECIMENS

Element	Concentration, ppm	
	As-Received Tubing	Tubing Removed from Boiler
O[a]	36, 41[b]	40, 41
N[a]	6, 7	7, 6
H[a]	2, 1	1, 1
C[c]	37, 38	43, 62

(a) Vacuum Fusion Analysis

(b) Duplicate Analysis

(c) Combustion Conductometric Analysis

TABLE II.

SPECTROGRAPHIC ANALYSIS OF 0.375-INCH T-111 TUBING

Element	Concentration in Acid Pickle Solution,[a] ppm			
	First Pickle		Second Pickle	
	As Received Tubing	Tubing Removed From Boiler	As Received Tubing	Tubing Removed From Boiler
Fe	38	102	35	< 20
Ni	6	26	< 5	< 5
Cr	17	22	< 10	< 10

(a) Entire specimen pickled, removing 0.001-inch thickness of material from both ID and OD per pickle.

3. Boiler Repair Plan

On August 23, 1968 NASA approval was received for the repair and reinstallation of the T-111 Corrosion Loop boiler. A number of modifications to the boiler design were required as shown schematically in Figure 4. Since one coil was removed from the boiler an additional length of T-111 one inch diameter tubing was added to the top of the boiler to achieve a total boiler height equal to the original boiler for correct fitup during installation into the loop. New fittings were required for attachment to the lithium inlet and outlet lines. During installation of the boiler the lithium lines presently on the loop will be inserted into socket fittings to insure correct alignment during welding. Butt welding is normally the technique utilized for joining tubing; however, because of the location of these welds and limited access during installation of the boiler into the loop the socket weld approach was selected. The main concern with this type of weld joint is the possibility of an open gap between the socket fitting ID and the OD of the inserted tubing. A trial fitting was machined with a double socket and weld experiments performed to develop a technique to prevent this gap. The welded specimen is shown in Figure 5. Subsequent radiographic and metallographic examination of this specimen indicated full penetration welds with no gaps were achieved when the tubing was inserted in the socket and pulled back slightly so the bottom of the tube did not contact the bottom of the socket. The joint between the 0.375-inch inner

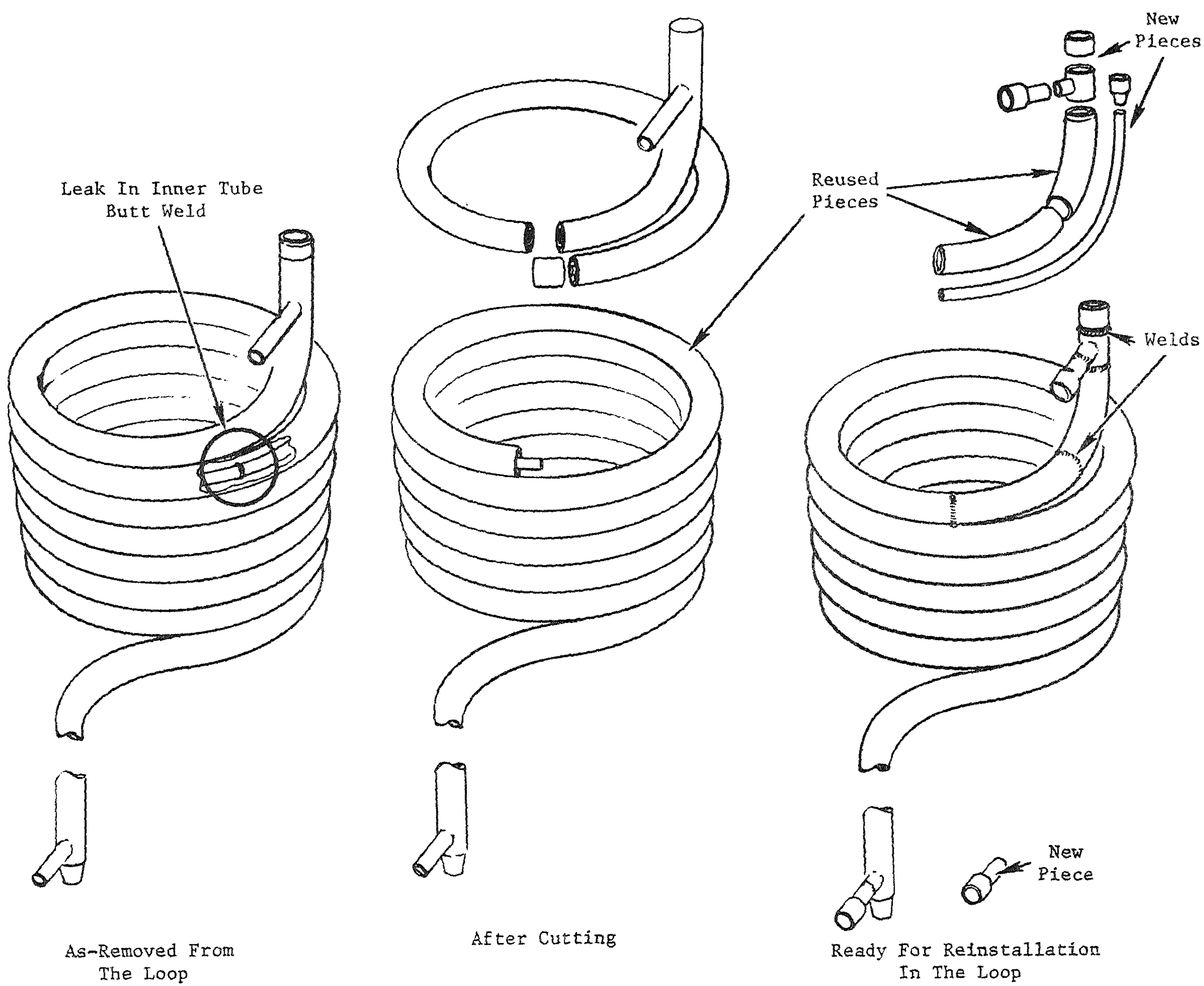
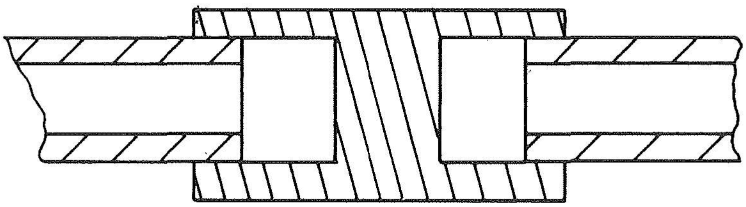
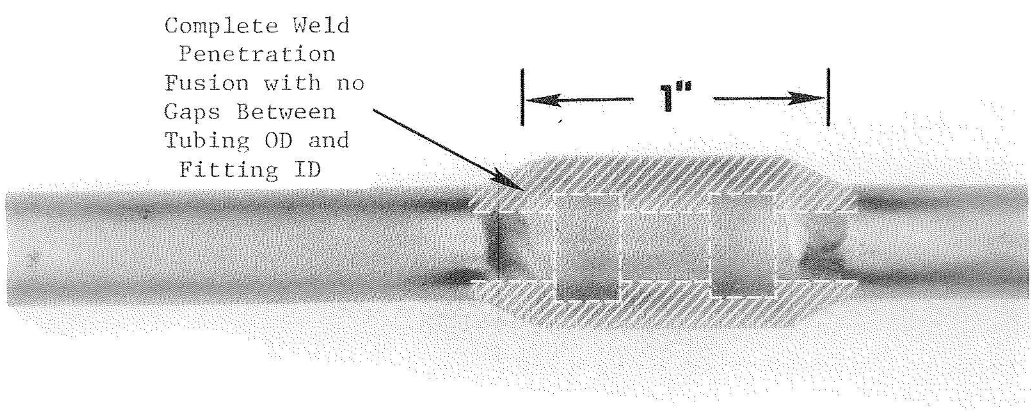


Figure 4. Schematic of T-111 Corrosion Loop Boiler Repair.



Before Welding



After Welding

Figure 5. Socket Weld Fitting Specimen to Qualify this Joint Design for T-111 Corrosion Loop Boiler Repair and Reinstallation. (C68082846)

boiler tube and the 1-inch outer boiler tube at the top of the boiler was also modified with a new fitting similar to that used at the bottom of the boiler. This butt joint design is preferred over the previously employed tube to header joint. These design modifications are compared with the original design in Figure 6.

4. Boiler Repair

The necessary machined parts were received and the boiler repair welding was initiated on August 29, 1968. Welding was performed in the VASCO welding chamber in the NSP Systems Materials Technology Laboratory according to NSP Specification 03-0025-00-A, "Welding of Columbium, Tantalum, and Their Alloys by the Inert Gas Tungsten Arc Process" (Appendix A).

In the initial welding step a new section of 0.375-inch-diameter tubing was butt welded to the inner boiler tube. Subsequent helium mass spectrometer leak checking and radiographic inspection indicated this weld to be sound. Two sections of 1-inch-diameter boiler tube were then welded in place as shown in Figure 7. These pieces were obtained from the boiler coil which was removed during sectioning of the boiler (Figure 4). Following the joining of the new end fitting to the 0.375-inch-diameter tubing the three welds were inspected by radiography and helium mass spectrometer leak checking and found to be sound. The boiler was completed with the addition of the lithium inlet fitting and boiler extension piece at the top of the boiler as shown in Figure 8, and the addition of the lithium outlet fitting at the bottom of the boiler as

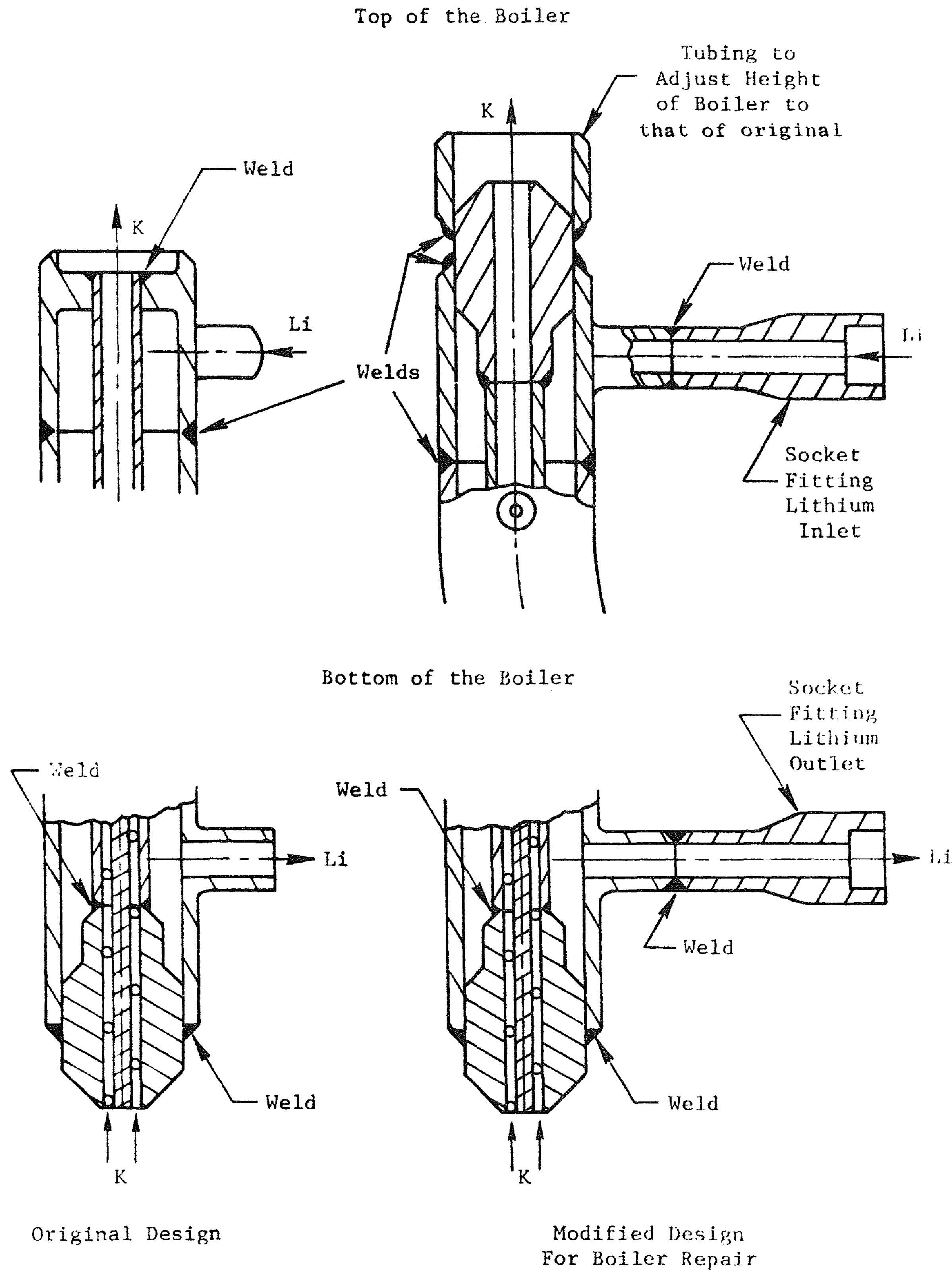


Figure 6. Joint Design at the Ends of the T-111 Corrosion Loop Boiler.

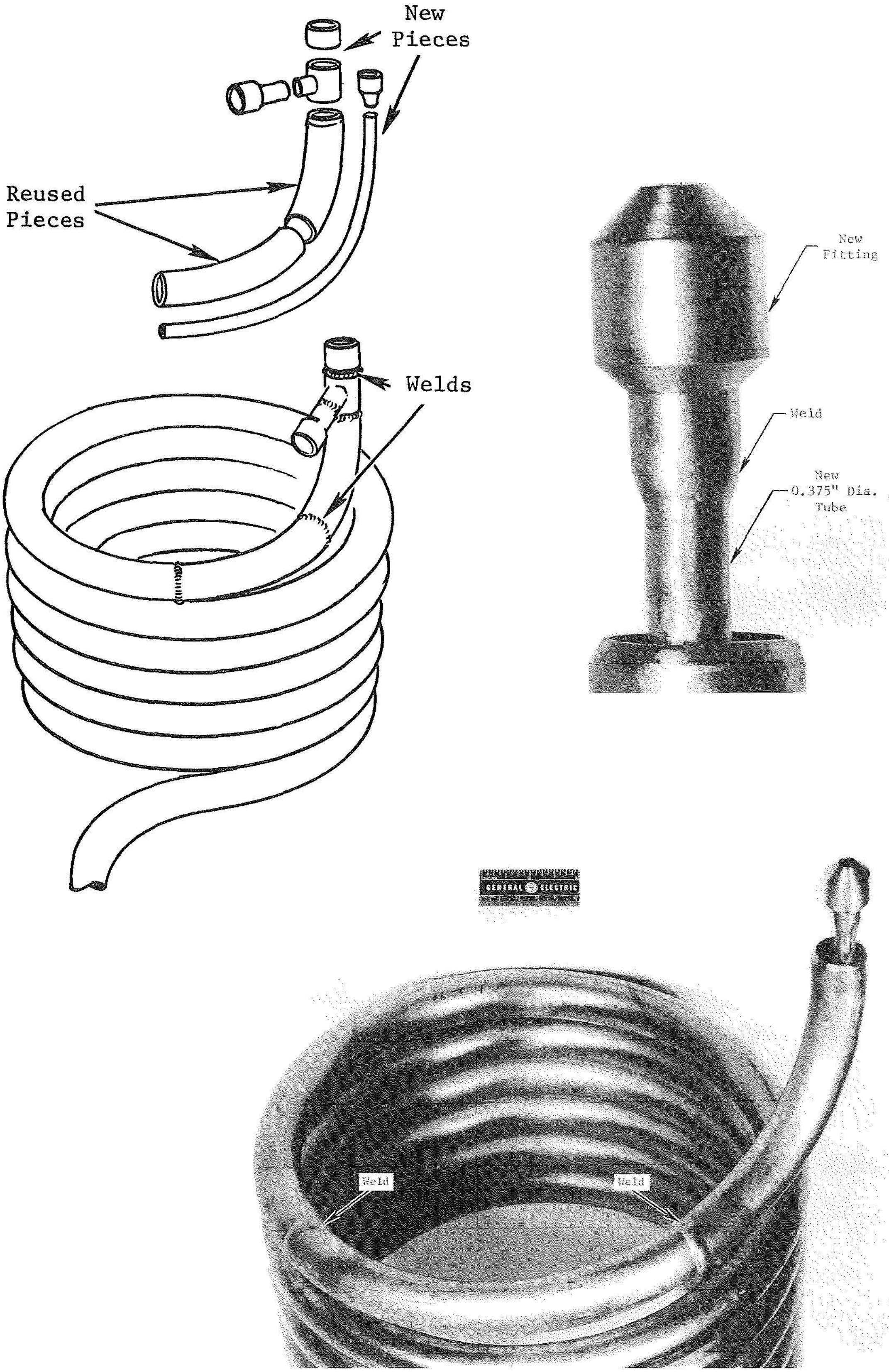
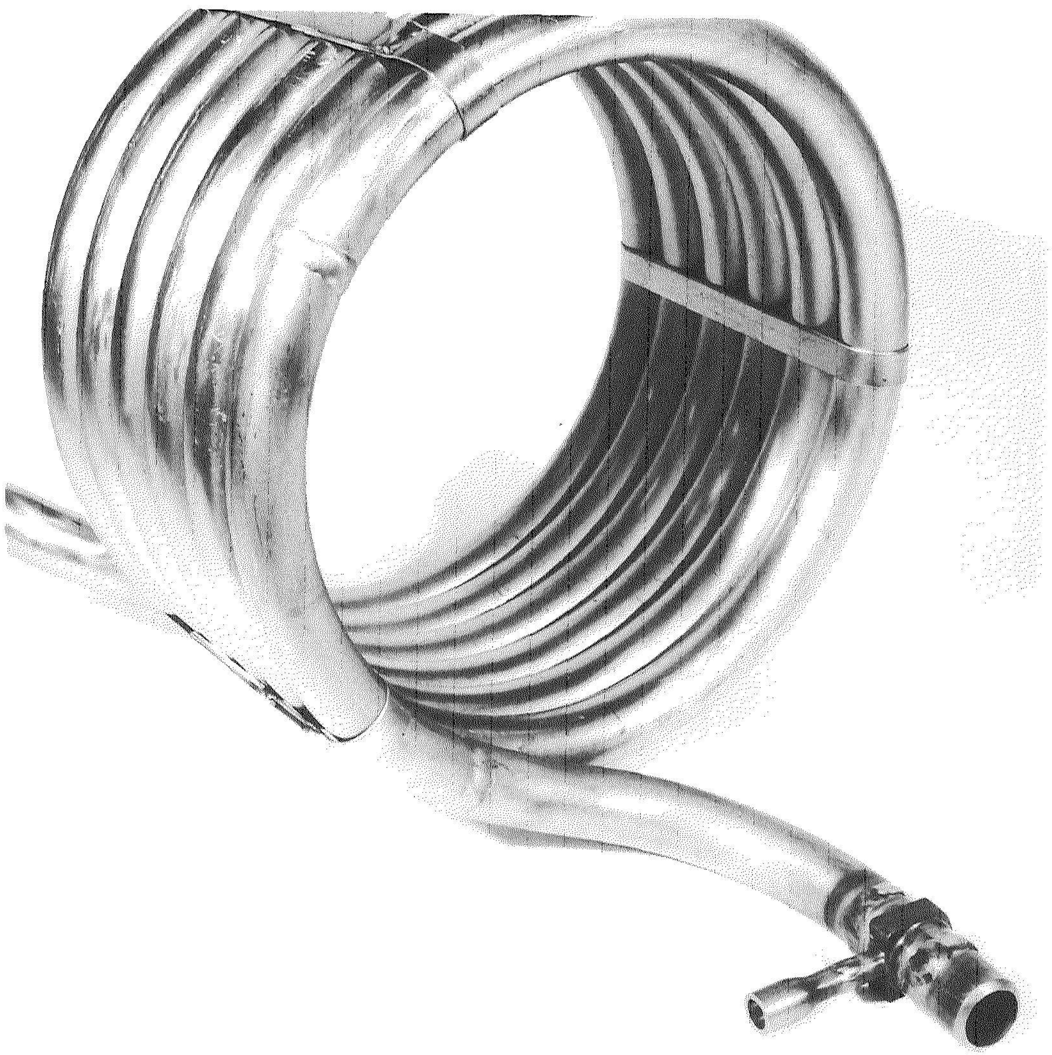


Figure 7. Top of the T-111 Boiler Following Initial Repair Welds. (Orig. P68-9-3B — Inset P68-9-3B)



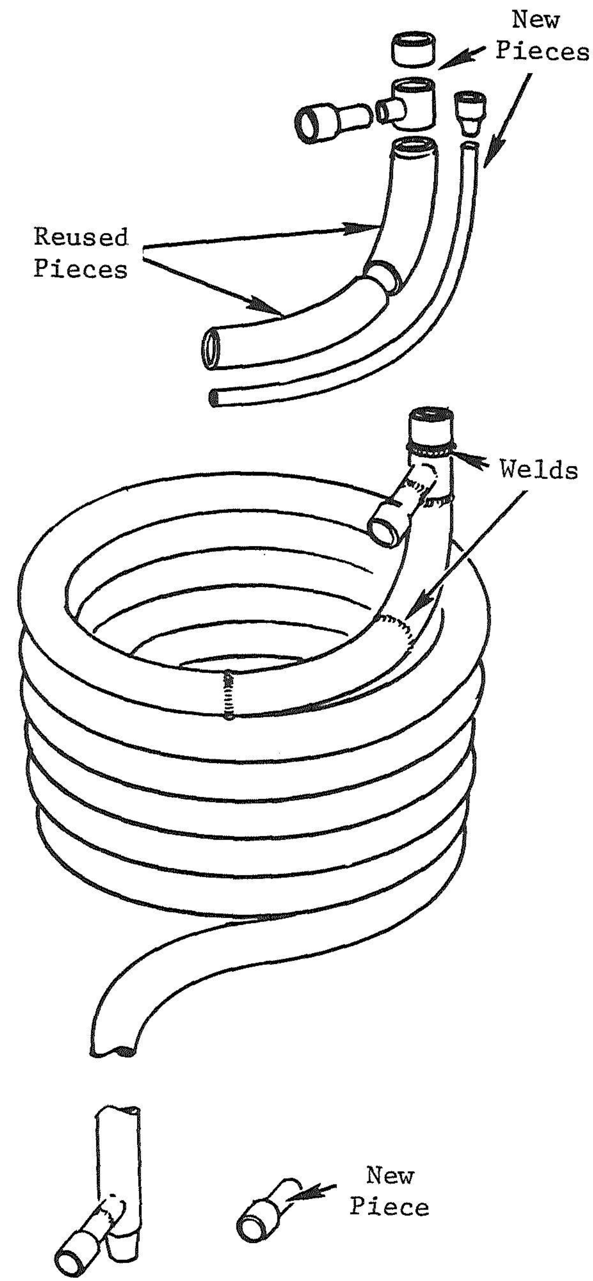


Figure 8. Top of T-111 Corrosion Loop Boiler Following Final Repair Welding. (Orig. P68-9-31B)

shown in Figure 9. The boiler plug was reattached at this time by tack welding to the bottom boiler fitting.

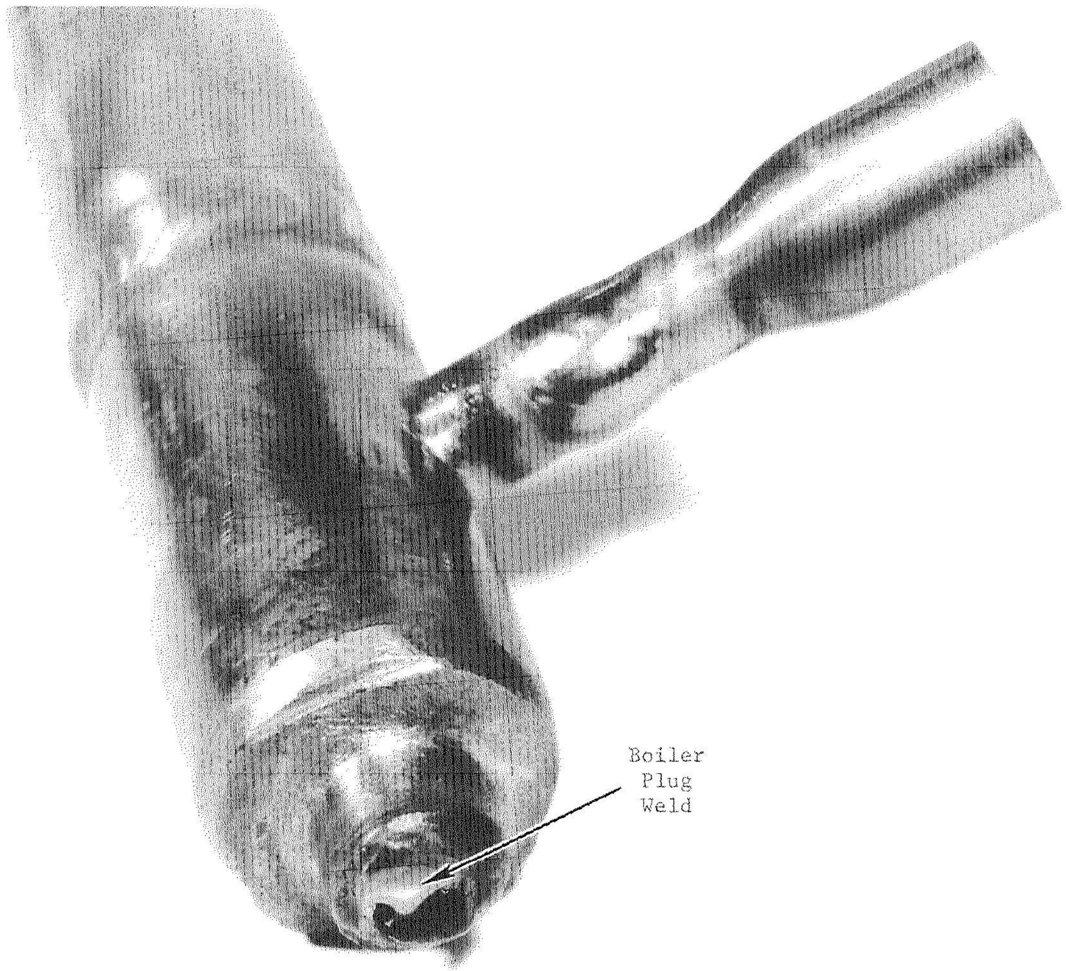
The repaired boiler is compared with the original boiler in Figure 10. The removal of one coil from the boiler will reduce its total length by approximately 27 inches; however, the performance of the repaired boiler should be similar since the overall length of the boiler is still greater than the length required to obtain the specified superheat (100°F).

A final helium mass spectrometer leak check was performed on the boiler to insure no leaks were present between the potassium and lithium circuits. No leak indications were observed.

5. Postweld Annealing of the Boiler

The repaired boiler was wrapped with Cb-1Zr foil as shown in Figure 11 in preparation for postweld annealing at Union Carbide Stellite Corporation, Kokomo, Indiana. A general description of the ABAR Model 90 furnace employed for this heat treatment is given in Table III. This chamber had been previously qualified on August 28, 1968 and the results of this qualification are given in Table IV.

The postweld anneal was performed successfully on September 10, 1968, according to NSP Specification 03-0037-00-A, "Postweld Vacuum Annealing of Cb-1Zr and T-111 Alloys." The chamber pressure was maintained below 1×10^{-5} torr at temperatures above 1000°F, and the maximum pressure with the boiler at 2400°F was only 5×10^{-6} torr. Analysis of T-111 coupons



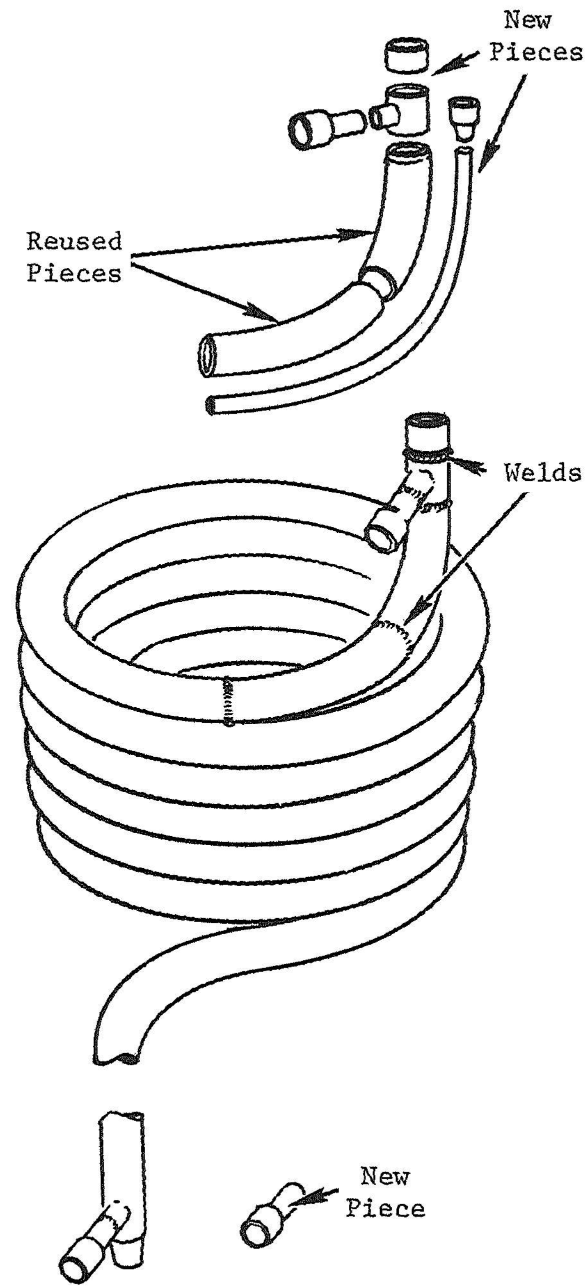
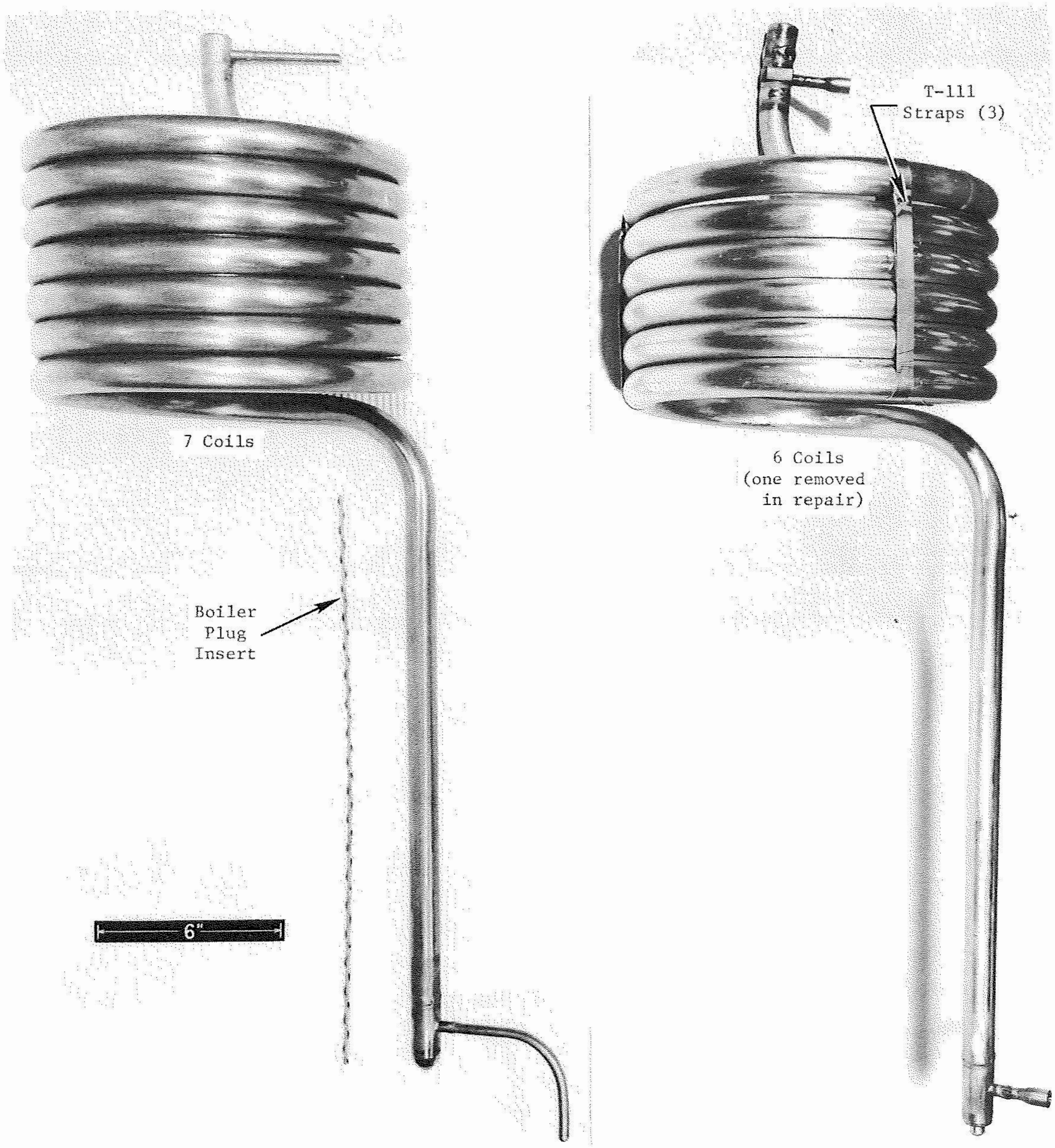


Figure 9. Bottom of the T-111 Boiler Following Repair Welding. (Orig. P68-9-31C)



Following Initial Fabrication

Following Repair

Figure 10. T-111 Corrosion Loop Boiler. (Orig. C67071832, P68-9-12H)

Figure 11. Repaired T-111 Corrosion Loop Boiler Wrapped with Cb-1Zr Foil in Preparation for Postweld Annealing. (C68090903)

TABLE III.

GENERAL DESCRIPTION OF THE ABAR MODEL 90 FURNACE
AT STELLITE UNION CARBIDE CORPORATION, KOKOMO, INDIANA

Cold Wall Furnace

Tantalum Strip Heater Elements

Tantalum Foil Reflective Insulation

Load Zone Dimensions: 23-inches Diameter x 39-inches High

Loading Method: Bottom loading

Maximum Temperature: 3200°F

Control: 1 heating zone

Pumping System:

- Mechanical: Kinney Model No. KDH130, 131 cfm
- Diffusion: 16-inch CVC, 10,000 liters/sec
- Baffle: CVC, Freon Refrigeration Cooled

Best Vacuum with Furnace Loaded: 1.2×10^{-6} torr

Leakup Rate: 0.3 micron/hour maximum after completion of run

TABLE IV.

QUALIFICATION OF UNION CARBIDE ABAR FURNACE
PERFORMED ON AUGUST 28, 1968

Chemical Analysis of 0.040-inch Thick Cb-1Zr Coupons

Element	Concentration, ppm		
	As-Received	Heat Treated[a]	
		Wrapped in Cb-1Zr Foil	Unwrapped
Nitrogen	21	25	14
Oxygen	36	41	34
Hydrogen	1	2	1
Carbon	45, 57	44, 45	36, 44

(a) 1 hour at 2500°F, maximum pressure 2×10^{-5} torr

TABLE V.

HEAT TREATMENT OF T-111 CORROSION LOOP BOILER

Chemical Analysis of 0.040-inch Thick T-111 Coupons

Element	Concentration, ppm		
	As-Received	Heat Treated[a]	
		Wrapped in Cb-1Zr Foil	Unwrapped
Nitrogen	9	9	9
Oxygen	83	88	87
Hydrogen	1	1	1
Carbon	33, 35	25,32	23,36

(a) 1 hour at 2400°F, maximum pressure at 2400°F 5×10^{-6} torr, ABAR Model 90 furnace Union Carbide Stellite Corporation, Kokomo, Indiana, September 10, 1968.

exposed along with the boiler are compared with that of as-received material in Table V and indicates no significant contamination occurred during the heat treatment.

The boiler was again helium mass spectrometer leak checked including across the circuits, with no indications observed.

6. Welding Chamber for the Installation of the Boiler into the Loop

Requirements to reinstall the boiler into the loop according to NSP Specification 03-0025-00-A, "Welding of Columbium, Tantalum, and Their Alloys by the Inert Gas Tungsten Arc Process" (Appendix A) necessitated the purchase of a special welding chamber to be installed around the loop for the welding operations. The stainless steel welding chamber was purchased from Vacuum Industries Inc., Somerville, Massachusetts. The chamber, shown during installation on the T-111 Corrosion Loop test facility in Figures 12 and 13, is comprised of two flanged spool sections four feet in diameter and four feet high such that each can be rotated independently for improved access to weld locations. Sight ports and glove ports are appropriately positioned in the areas where welding will be performed at the top and bottom of the boiler location. An independently pumped tool port is also provided such that necessary tools could be brought into the chamber without contaminating the chamber environment.

The chamber is shown in Figure 14 after final installation. Rough pumping was accomplished with the 260 liter per second turbomolecular

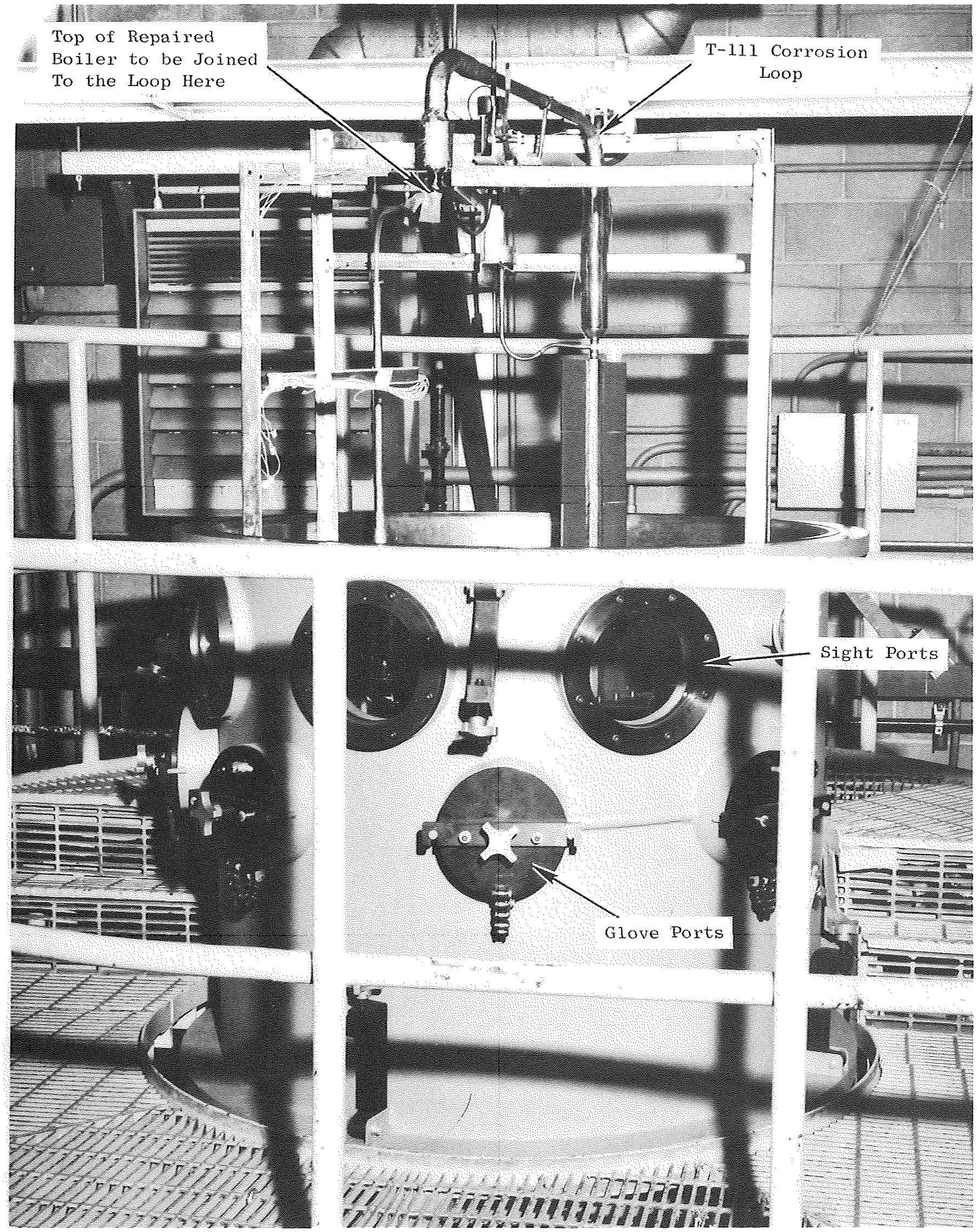


Figure 12. Installation of the Welding Chamber Around the T-111 Corrosion Loop. Lower Spool Piece in Position. (C68090429)

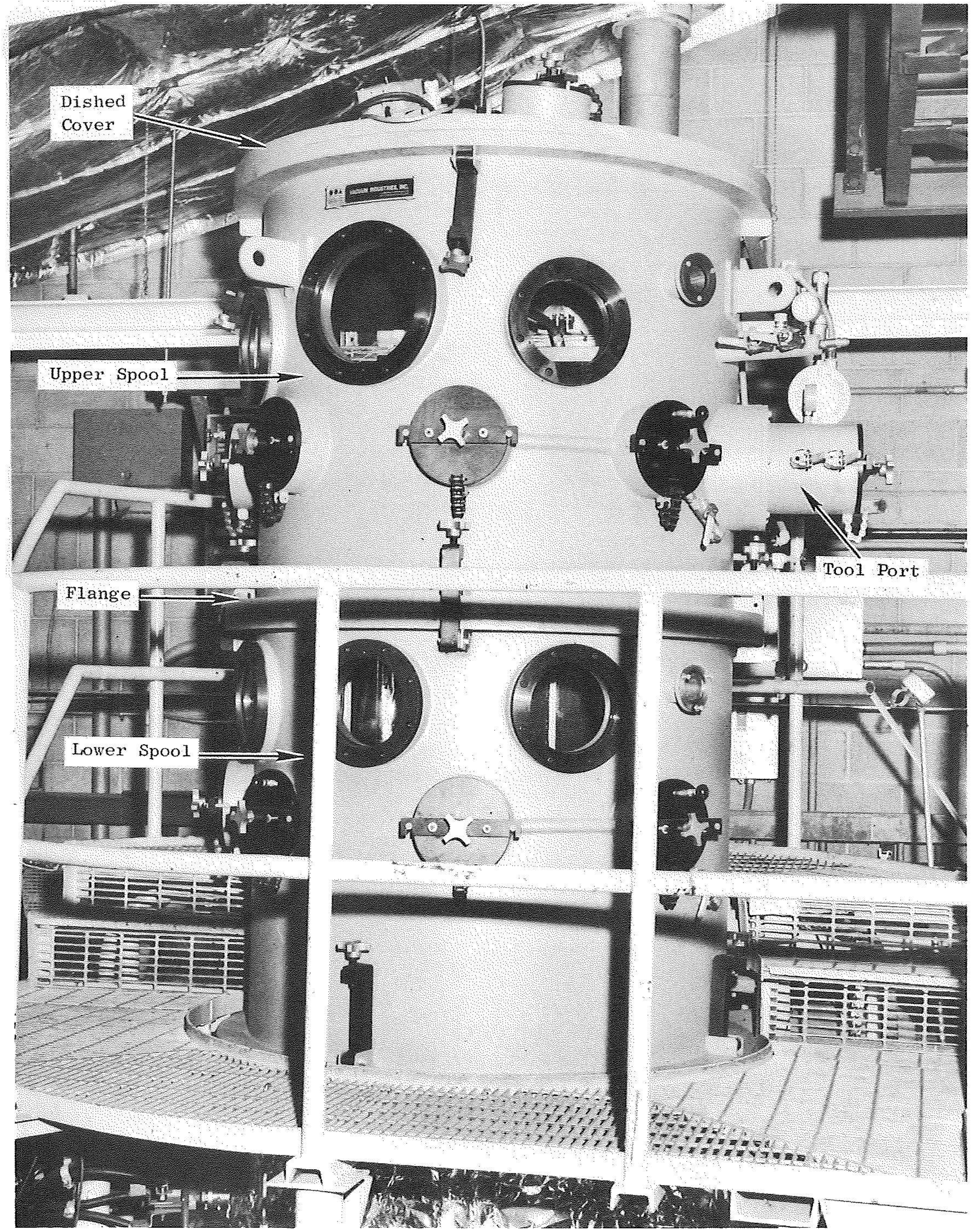


Figure 13. Welding Chamber Installed Around the T-111 Corrosion Loop. The Chamber Consists of Two Spools Which Can Be Rotated Independently for Improved Access to Weld Locations. (C68090424)

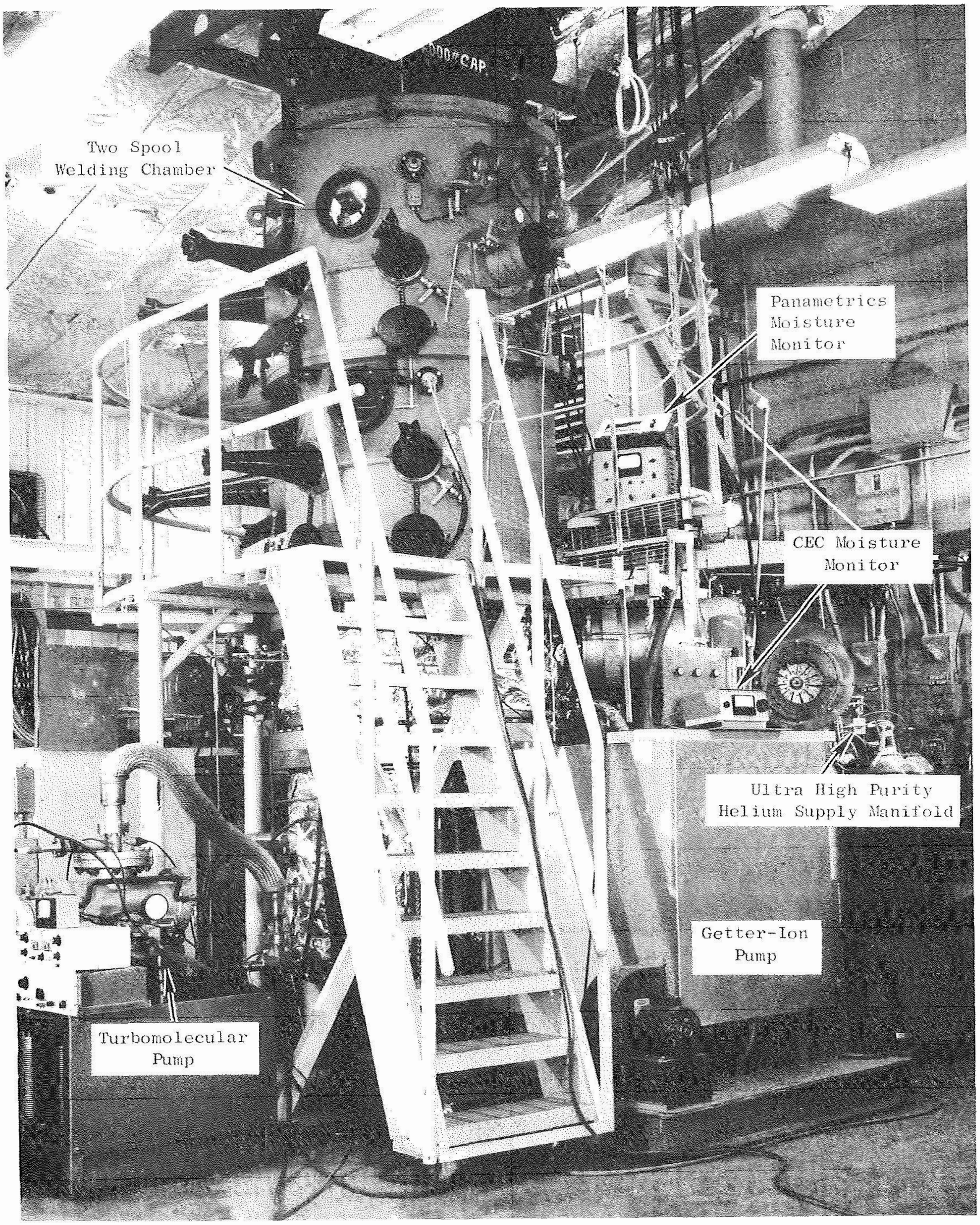


Figure 14. Welding Chamber and Ancillary Instrumentation Installed on the T-111 Rankine System Corrosion Test Loop Facility. (P68-9-44C)

pump, and the loop facility ion pumps were used to achieve the high vacuum ($< 1 \times 10^{-5}$ torr) called for in the welding specification. The chamber was backfilled with ultra-high purity helium which was passed through a molecular sieve dryer before entering the chamber. The inert gas analysis equipment included C.E.C.* and Panametrics** moisture monitors and the gas chromatograph shown in Figure 15. Gas lines attached to the gas chromatograph made analysis of the inlet gas as well as outlet gas from the chamber possible.

Before welding of the boiler could be initiated the chamber and welding equipment had to be qualified according to Section 4.3 of the forementioned welding specification. The chamber was qualified for use on September 21, 1968 and the results of that qualification are presented in Appendix B.

7. Installation of the Boiler into the Loop

The boiler was welded into the loop on September 27, 1968 as shown in Figure 16. Four welds were required to reinstall the boiler; two at the top of the boiler and two at the bottom as shown in Figures 17 and 18, respectively. Subsequent radiographs of the welds indicated a very small area in the upper lithium line weld of incomplete penetration. Although the weld was acceptable from a joining standpoint a decision was made to reweld the joint to improve reliability. The chamber was reassembled on the loop facility and welding completed on October 2, 1968. Subsequent mass spectrometer helium leak checking and radiography of the welds

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*Model 26-303 Consolidated Electrodynamics Corp., Cleveland, Ohio

**Model 1000 Panametrics Inc., Waltham, Mass.

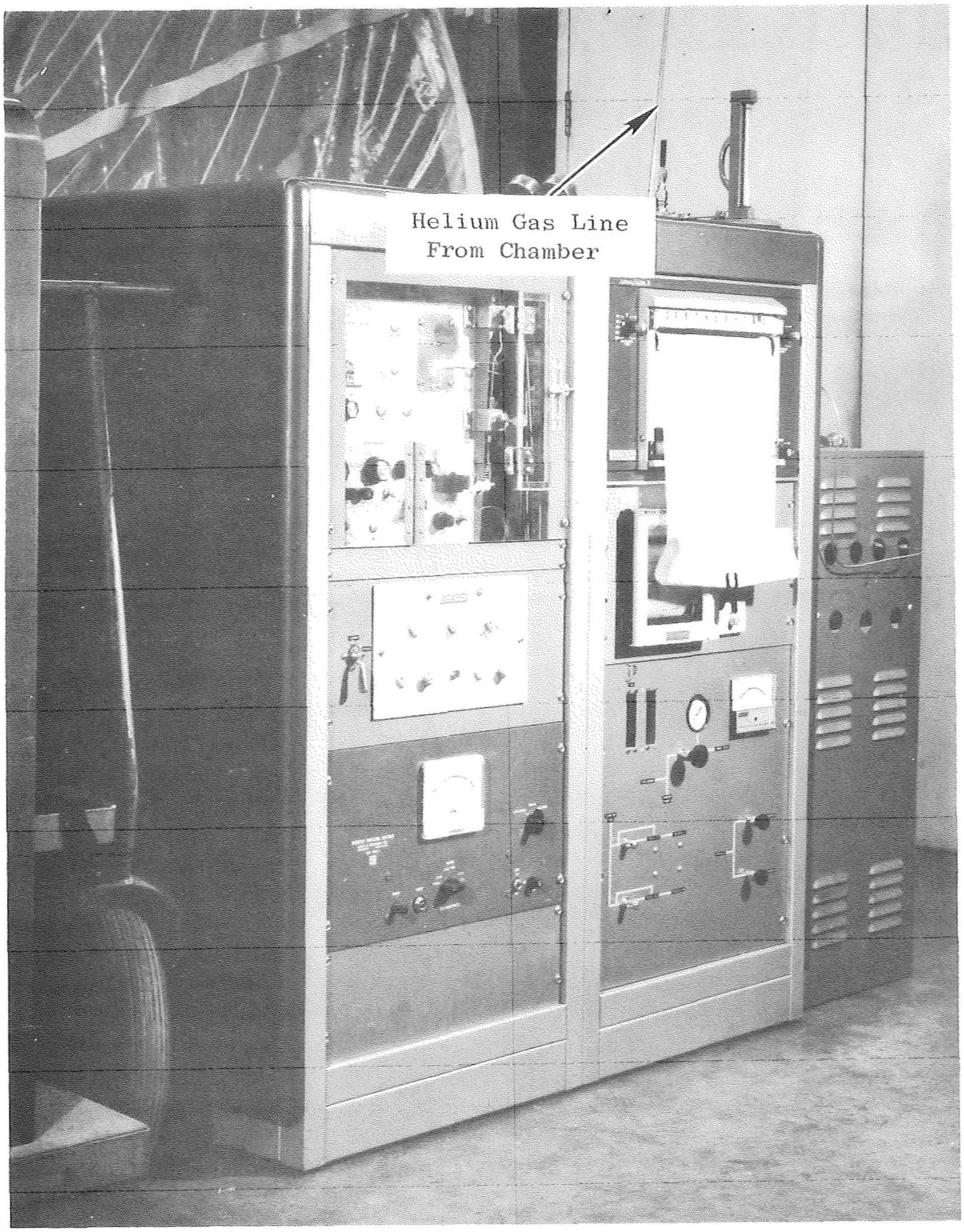

Figure 15. Gas Chromatograph Used to Analyze the Helium Atmosphere in the Welding Chamber for the Repair of the T-111 Corrosion Loop. (P68-9-44B)

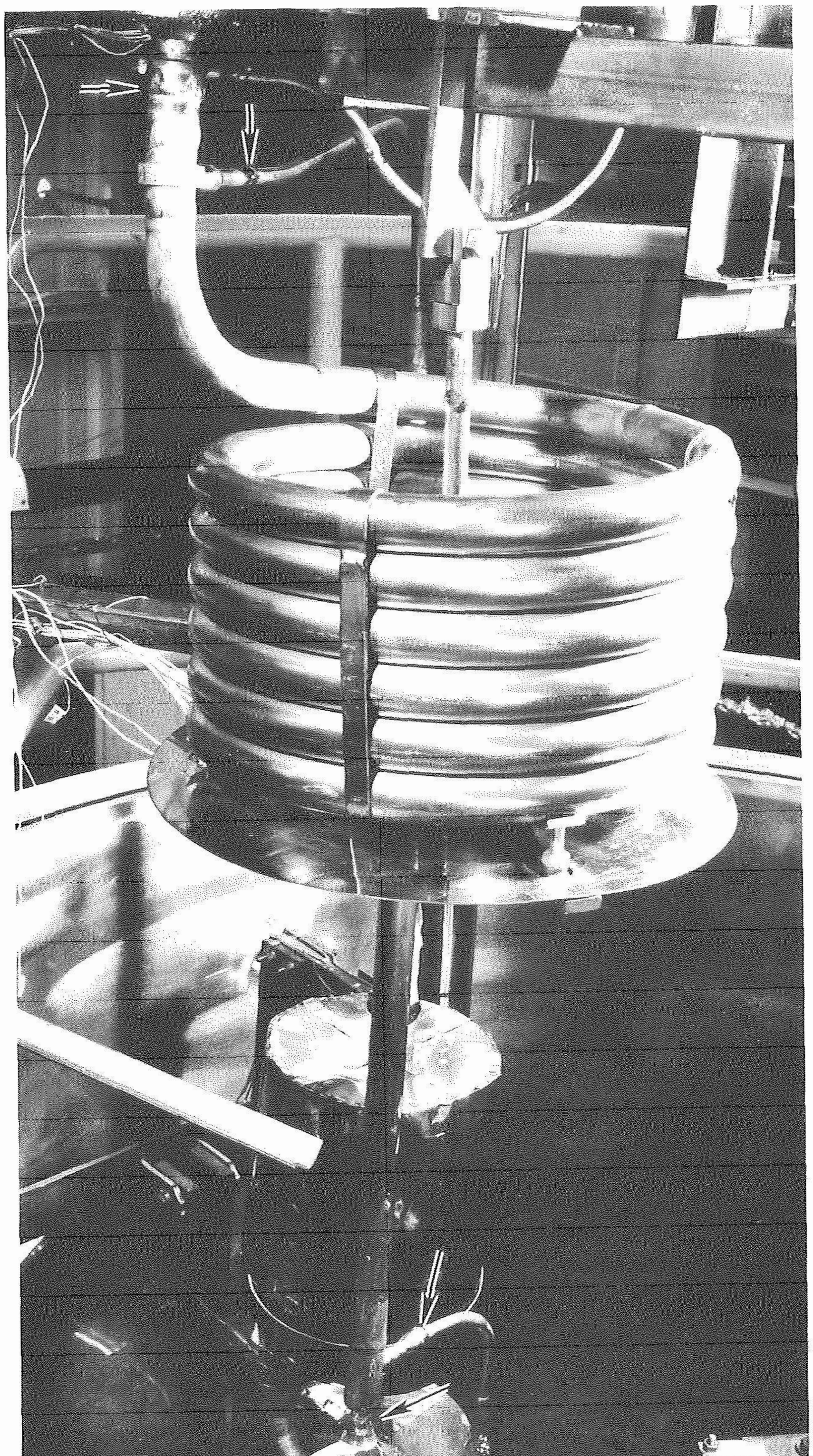

Figure 16. Repaired Boiler Installed into the T-111 Corrosion Loop. Arrows Indicate Installation Welds. (P68-10-37B)

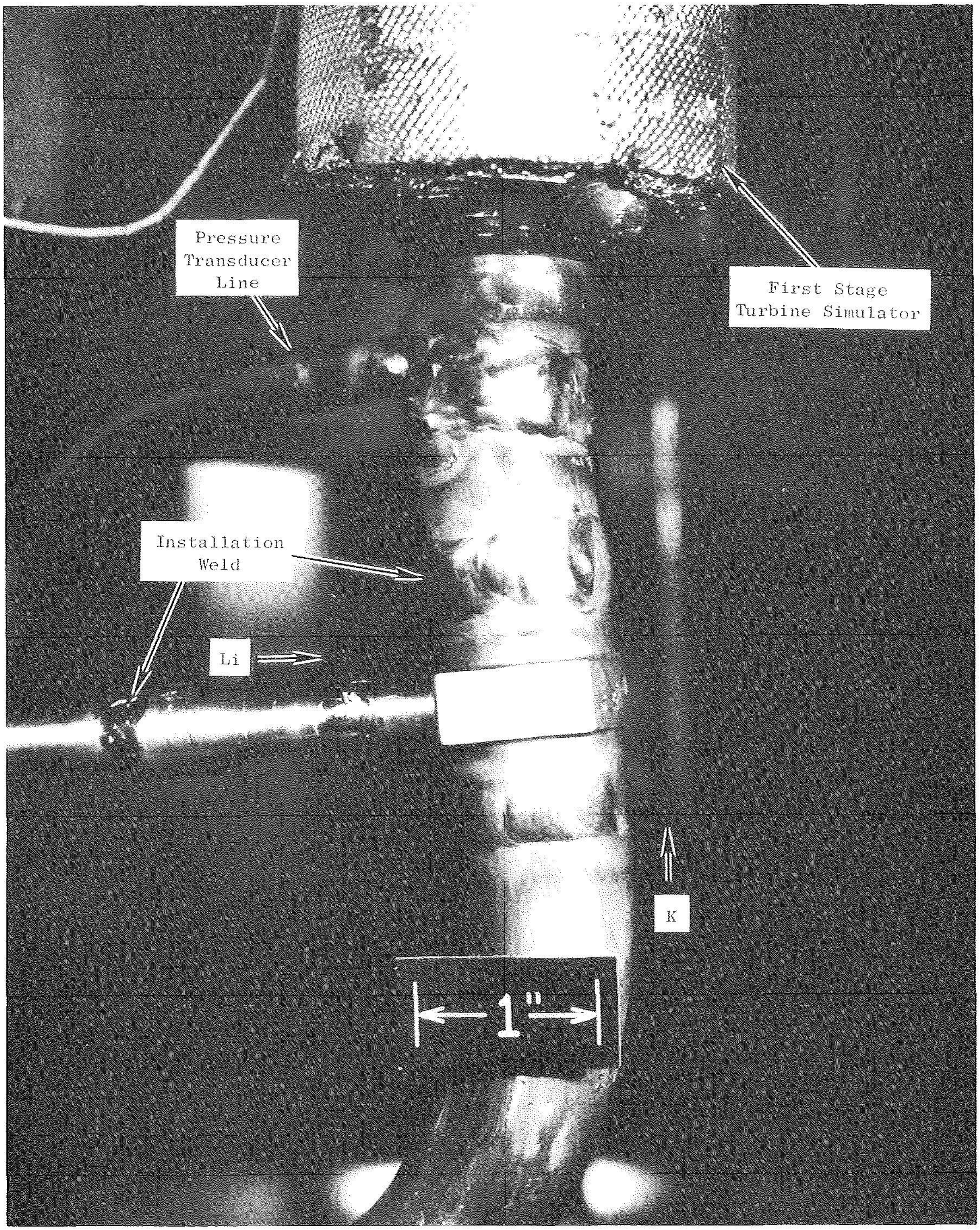

Figure 17. Top of Repaired Boiler After Installation in the T-111 Corrosion Loop. (P68-10-37A)

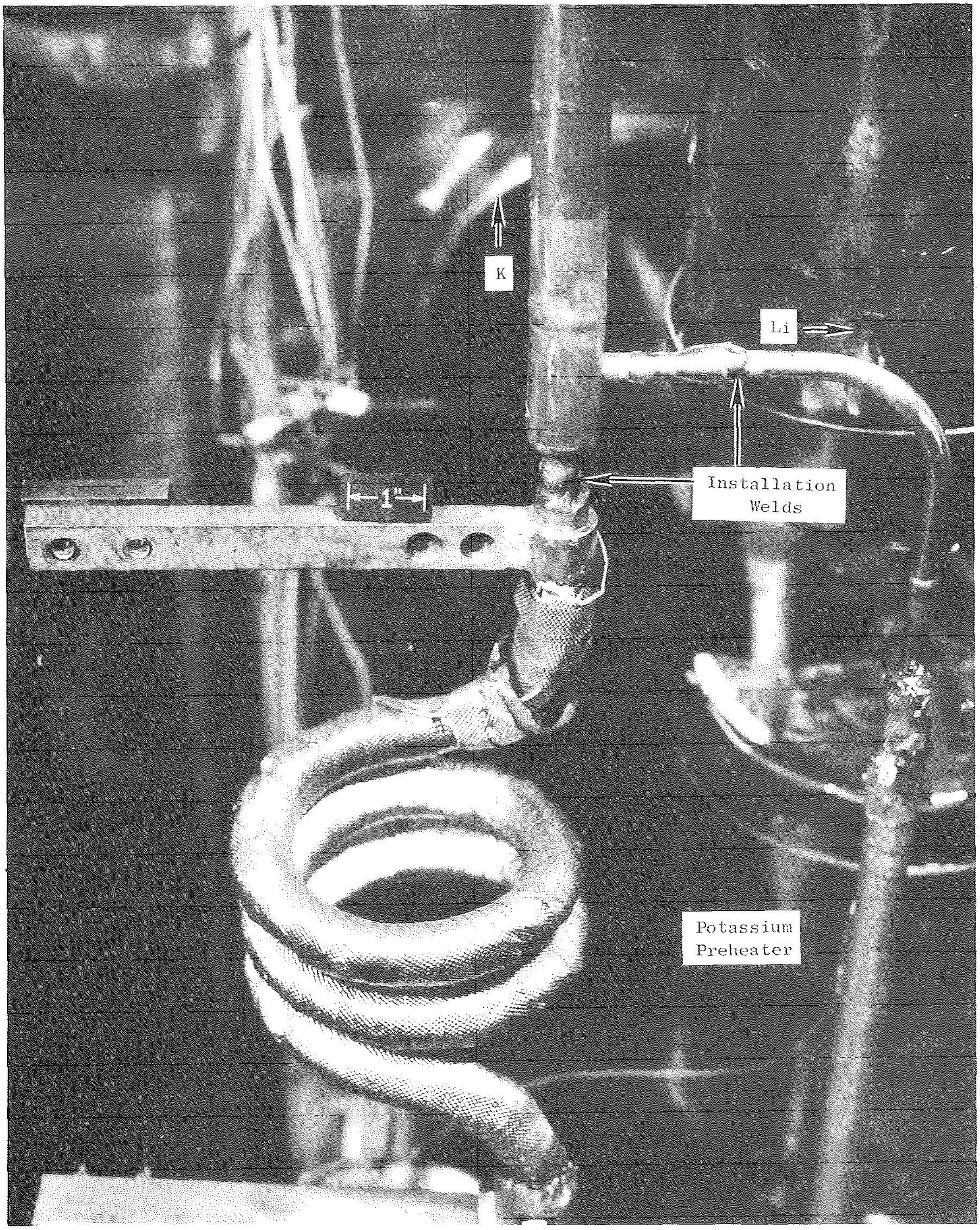

Figure 18. Bottom of Repaired Boiler After Installation in the T-111 Corrosion Loop. (P68-10-37C)

showed no indications of leaks or weld defects. The upper spool piece of the chamber was removed and installation of the annealing furnaces around the welds was initiated. A typical annealing furnace is shown in Figure 19 and is comprised of tantalum shields and shell, alumina insulators, and tungsten wire (0.050" Dia.) elements. The power for heating the elements was supplied from a standard welding machine, one electrode being the welding torch and the other electrode grounded. The welds were annealed for 1 hour at 2400°F. The chamber was opened on October 15, 1968 and subsequent mass spectrometer leak checking performed. No indications were noted including a leak check between the potassium and lithium circuits.

8. Alkali Metal Purification and Control

As reported previously[1] spectrographic analysis of recently hot trapped and distilled lithium indicated higher than normal concentrations of iron, nickel and chromium, and also high concentrations of aluminum and silicon. It was conjectured that the source of the iron, chromium and nickel was the sintered stainless steel filter through which the distilled lithium had been filtered. Consequently, this filter was removed, samples being taken before (No. 1831) and after (No. 1887) its removal. The analytical results for these two samples are shown in Table VI along with the results for previous samples. Samples 1806 and

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[1] Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 13 for Period Ending July 15, 1968, NASA Contract NAS 3-6474.

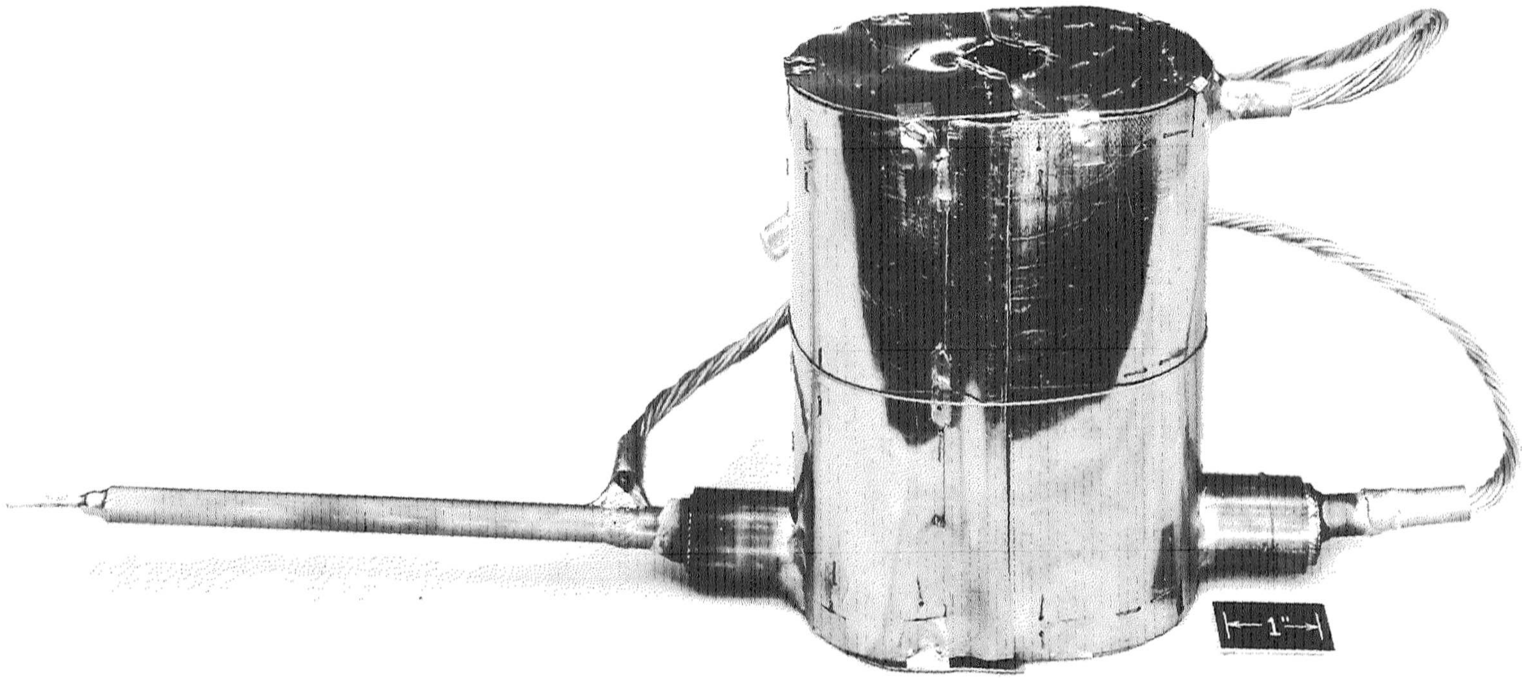

Figure 19. Refractory Metal Furnace for Annealing of Corrosion Loop Weldments. (C65032922)

TABLE VI.

ANALYSIS OF DISTILLED LITHIUM

Sample No.	Concentration, ppm							
	Sampled Through Filter[a]						Sampled Directly[b]	
	1806 A		1806 B[c]		1831		1887	
Element	GE	NSL[d]	GE	NSL	GE	NSL	GE	NSL
O								
C	53,33 88						67	
N							17,24	
Ag	< 5	ND		ND	< 5	ND	5	ND
Al	125	185		65	50	165	5	< 25
B	< 50	ND		ND	< 75	ND	< 50	ND
Ba	< 75	ND		ND	<25	< 500	< 50	ND
Be	< 5				< 5		< 5	ND
Ca	25	95		50	< 5	35	< 5	20
Cb	< 25	ND		ND	< 5	ND	< 25	ND
Co	< 5	ND		ND	< 5	ND	< 5	ND
Cr	25	280		120	< 5	< 25	< 5	< 25
Cu	5	60		< 5	25	< 5	5	< 5
Fe	< 5	120		25	< 5	15	25	20
K		< 50		T		< 500		
Mg	< 5	< 50		T	< 5	< 500	< 5	< 500
Mn	< 5	ND		ND	< 5	ND	< 25	ND
Mo	< 5	ND		ND	< 5	ND	< 5	ND
Na	< 75	< 50		T	75	< 500	< 50	< 500
Ni	< 5	700		280	< 5	< 25	< 5	< 25
Pb	< 75	< 100		ND	< 75	ND	< 75	ND
Si	> 125	700		600	> 125	275	< 25	< 25
Sn	< 25	ND		ND	< 25	ND	< 25	ND
Sr	5	< 50		ND	5	ND	< 5	ND
Ti	< 25	ND		ND	< 5	ND	< 5	ND
V		ND		ND	< 25	ND	< 25	ND
W		ND		ND		ND		ND
Zn		ND		ND		ND		ND
Zr	< 25	ND		ND	< 25	ND	< 25	ND

(a) Sintered stainless steel filter

(b) Filter removed

(c) Separate aliquot of lithium used for sample 1806 A

(d) National Spectrographic Laboratory

1831 were both from the same batch of lithium removed from the still receiver through the stainless steel filter and consequently the analytical results should be comparable. The results are similar with respect to the values for aluminum and silicon; but the values for iron, chromium and nickel are much reduced in sample 1831. The removal of the filter appears to have resulted in reducing the concentrations of aluminum and silicon. The concentrations of metallic impurities in sample 1887 are sufficiently low to make it acceptable for use. In spite of the fact that the first sample taken without the filter was of acceptable purity, all the lithium was returned to the hot trap for redistilling and subsequent analysis for metallic constituents. Distillation of this lithium was completed and a sample taken. The analysis of this sample (No. 1925) of the redistilled lithium is compared with that of a similar sample taken before redistillation (No. 1887) in Table VII. Both samples were taken without passage through a filter. One additional sample was taken from the receiver for final qualification of the purification facility and procedure. Because of prior indications of particulate matter in distilled lithium,(1) high purity argon was admitted to the receiver through the dip leg in order to agitate the lithium. This technique should produce a sample containing particulate matter if any was present. The sample (No. 1986) was taken immediately after agitation. The analysis

(1) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 13 for Period Ending July 15, 1968, NASA Contract NAS 3-6474.

TABLE VII.

ANALYSIS OF DISTILLED LITHIUM[a]

	Concentration, ppm			
	Initial Distillation		Lithium Returned to Hot Trap and Redistilled[c]	
Sample No.	1887		1925	1986
Analytical Lab.	GE	NSL[b]	GE	GE
Element				
O			9, 10	
C	67		30, 55	60
N	17,24		8, 9	17,20
Ag	< 5	ND	< 5	< 5
Al	5	< 25	< 5	25
B	< 50	ND	< 10	< 50
Ba	< 50	ND	< 10	
Be	< 5	ND	< 5	< 5
Ca	< 5	20	< 5	25
Cb	< 25	ND	< 25	< 25
Co	< 5	ND	< 5	< 5
Cr	< 5	25	< 5	< 5
Cu	5	5	25-50[d]	5-25[d]
Fe	25	20	< 5	5
K				
Mg	< 5	< 500	< 5	25
Mn	< 25	ND	< 5	25
Mo	< 5	ND	< 5	< 5
Na	< 50	< 500	< 50	< 50
Ni	< 5	< 25	< 5	< 5
Pb	< 75	ND	< 75	< 25
Si	< 25	< 25	< 1	50
Sn	< 25	ND	< 5-50[d]	< 25
Sr	< 5	ND	< 5	
Ti	< 5	ND	< 5	< 5
V	< 25	ND	< 50	< 25
W		ND		
Zn		ND		
Zr	< 25	ND	< 25	< 25

(a) Both samples taken without passage through a filter

(b) National Spectrographic Laboratory

(c) Lithium in still receiver agitated with argon gas before sampling.

(d) Duplicate spectrograms made for tin and copper.

of this sample is shown in Table VII. Although slight increases in concentration of some impurities were noted in this sample (No. 1986) these increases were not considered to be significant.

Subsequently it was discovered that during the agitation of the lithium with argon some lithium was inadvertently blown back into the vacuum manifold on the lithium purification system. The vacuum manifold will be disconnected from the receiver, cleaned, and reinstalled before distillation of lithium for the loop is initiated.

The hot trap was filled with 28 pounds of lithium from the shipping container. The lithium was purchased from Foote Mineral Co., Exton, Pa. The sample obtained during filling contained 278 ppm N. Additional analysis of this sample is being obtained and will be reported in the next interim report. Subsequently the hot trap was heated to 1500°F. The lithium will be hot trapped for approximately 300 hours at which time additional analysis will be obtained.

The potassium hot trap was filled with 39.5 pounds of potassium from the shipping container. The potassium was purchased from Mine Safety Appliances Research Corporation, Callery, Pennsylvania. The hot trap was subsequently heated to 1300°F and held at that temperature for 30 hours. The potassium was further purified by vacuum distillation at 600°F. The analysis of the potassium after distilling is compared with that for the as-received material in Table VIII. The purified potassium

TABLE VIII

ANALYSIS OF POTASSIUM

Element	Concentration, ppm	
	As-Received	Hot Trapped and Vacuum Distilled
O	23	6
C	83	29
Ag	< 2	< 2
Al	2	< 2
B	< 30	< 20
Ba	< 30	< 20
Be	< 2	< 2
Ca	< 2	< 2
Cb	< 10	< 10
Co	< 2	< 2
Cr	< 2	< 2
Cu	< 2	< 2
Fe	< 2	< 2
Mg	< 2	< 2
Mn	< 2	< 2
Mo	< 2	< 2
Na	< 30	< 20
Ni	< 2	< 2
Pb	< 30	< 10
Si	2	< 2
Sn	< 10	< 10
Sr	< 2	< 2
Ti	< 10	< 10
V	< 10	< 10
Zr	< 10	< 10

will be stored under argon at room temperature until the loop is ready for flushing and filling with alkali metals.

9. Lithium-Potassium Solubility Study

As previously reported[2] examination of the alkali metals drained from the loop indicated lithium in the potassium and potassium in the lithium as a result of the boiler leak. Particulate matter was also found in the potassium. The particles and contaminated alkali metals will be removed from the loop by repeatedly flushing the circuits with alkali metals. The success of cleaning the loop in this manner is dependent on the mutual solubilities of lithium and potassium as a function of temperature. Limited data in the literature indicates these metals do not alloy.[3] Therefore, a study has been initiated to determine the mutual solubilities of lithium and potassium in the temperature range of 400°F to 1200°F.

A schematic diagram of the solubility apparatus is shown in Figure 20. The apparatus will be brought to a specific temperature and held for at least 24 hours to allow equilibration of the alkali metal solutions. Samples of lithium and potassium will be taken at the temperature of interest. The entire sample must be analyzed to determine the total lithium concentration in potassium or potassium in lithium since the solubility will change during cooling of the sample. A special sampling

(2) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 12 for Period Ending April 15, 1968, NASA Contract NAS 3-6474, NASA-CR-72452.

(3) Hansen, M., Constitution of Binary Alloys, McGraw Hill, New York, 1958, page 875.

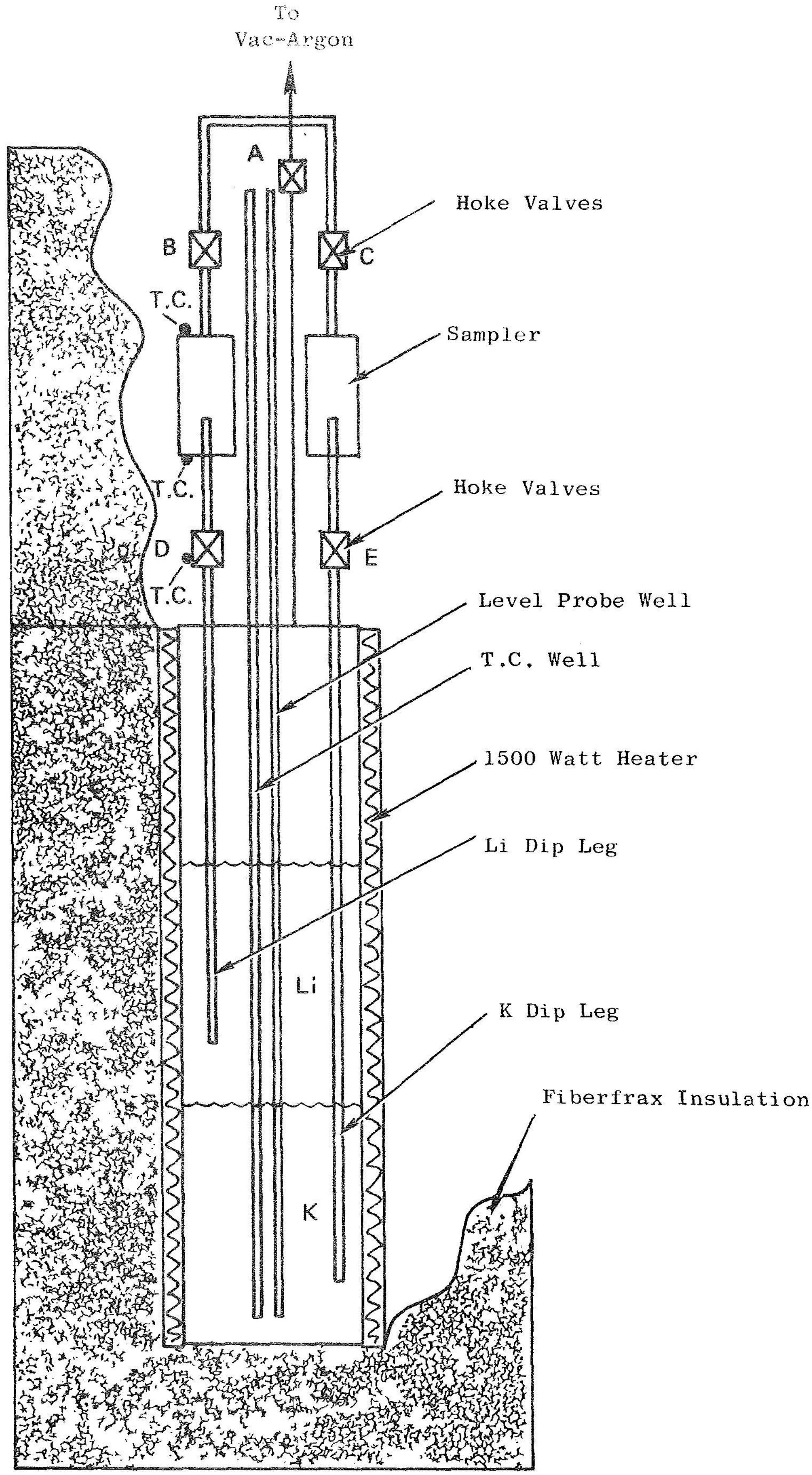

Figure 20. Lithium-Potassium Solubility Apparatus.

device will be employed to accomplish this requirement. The sampler, shown in Figure 21, will be filled with the alkali metal and drained leaving the representative sample in the cavity (approximately 7 cc volume) for subsequent analysis.

The apparatus has been filled with equal volumes of lithium and potassium which had been previously purified by vacuum distillation. Solubility data will be obtained at 600°F, 800°F, 1000°F, and 1200°F during the next month.

B. ADVANCED TANTALUM ALLOY CAPSULE TESTS

Testing of two ASTAR 811C and one ASTAR 811CN lithium thermal convection capsules was initiated. The capsule test facility is shown in Figure 22 just before closing the bell jar. As of October 15, 1968 the capsules completed 230 hours of testing at 2400°F. The chamber pressure at that time was 5×10^{-7} torr.

C. 2600°F LITHIUM LOOP

Final assembly of the 2600°F Lithium Loop will be accomplished upon completion of the lithium heater subassembly. This subassembly cannot be made until the tensile and corrosion test specimens of the ASTAR alloys are heat treated at conditions to be specified by the NASA Program Manager.

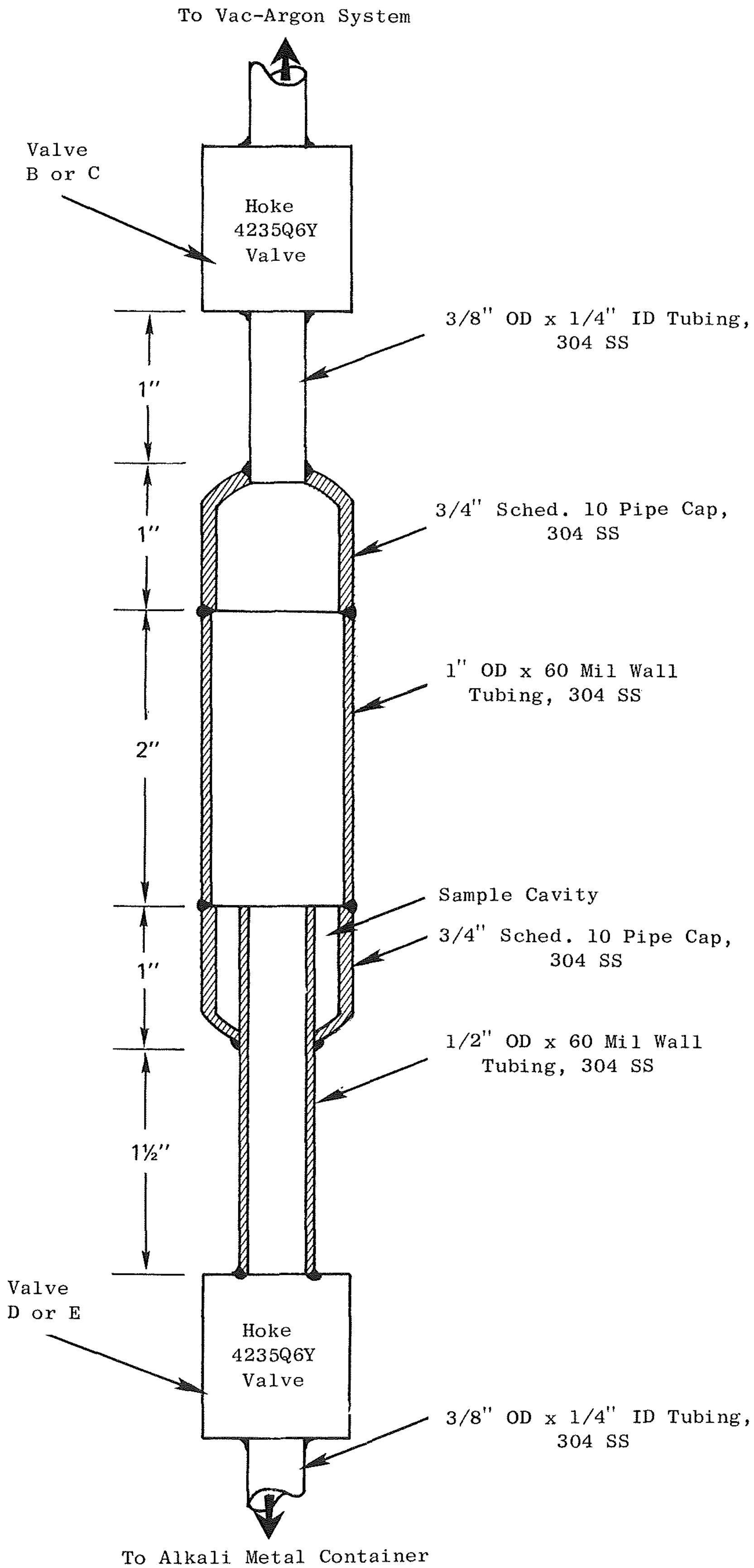

Figure 21. Lithium-Potassium Solubility Study Sampler.

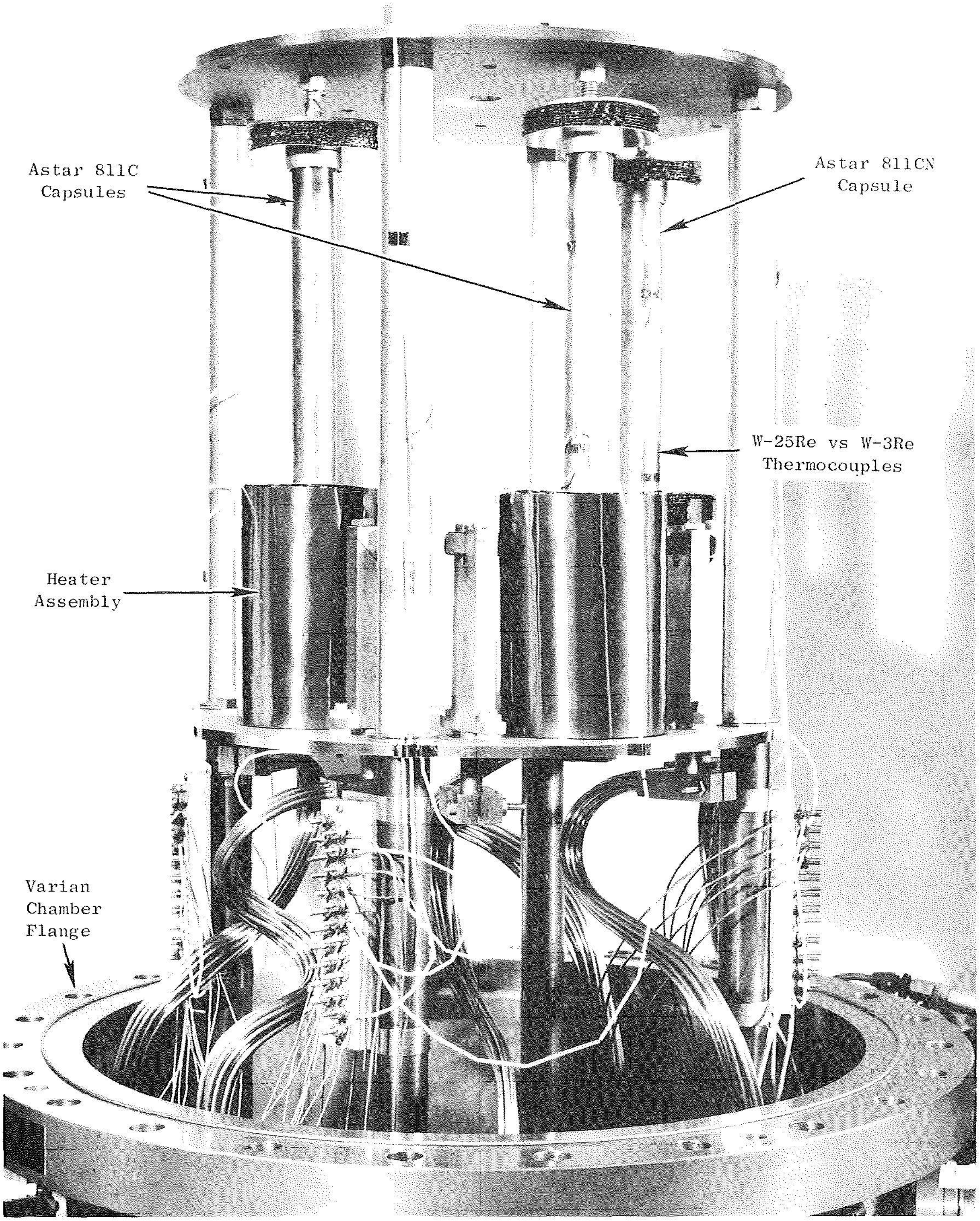

Figure 22. Lithium Thermal Convection Capsule Test Facility. Varian 18-Inch Diameter Chamber Capable of Cold Wall Vacuums of Less than 1 x 10^{-9} Torr. (P68-9-55)

IV. FUTURE PLANS

A. Complete reinstrumentation of the T-111 Corrosion Loop.

B. Complete the purification of alkali metals.

C. Determine the mutual liquid solubilities of lithium and potassium as a function of temperature.

D. Clean the loop circuits by flushing with alkali metals.

E. Fill the loop and initiate operation.

F. Continue advanced tantalum alloy capsule tests.

APPENDIX A

NSP Specification 03-0025-00-A Welding of Columbium, Tantalum, and Their Alloys By the Inert Gas Tungsten Arc Process

GENERAL ELECTRIC Nuclear Systems Programs Cincinnati, Ohio 45215	SPECIFICATION NO. 03-0025-00-A
ENGINEERING SPECIFICATION	DATE 1-26-66

TITLE	ORIGINAL CONTRACT
WELDING OF COLUMBIUM, TANTALUM, AND THEIR ALLOYS BY THE INERT GAS TUNGSTEN ARC PROCESS	

1. SCOPE

1.1. Scope. This specification establishes the procedures, process substantiation, and quality requirements for fusion welding columbium, tantalum, and their alloys by the inert gas shielded tungsten arc-welding process. Reference to this specification shall be made on engineering drawings whenever applicable. Information or requirements on the drawings supersede this specification.

2. APPLICABLE DOCUMENTS

2.1. Government Documents. None

2.2. Non-Government Documents

ASTM B297-55T (1955)	Tentative Specification for Tungsten Arc-Welding Electrodes (AWS A5.12-55T)
ASTM E142-64 (1964)	Controlling Quality of Radiographic Testing
ASTM E94-62T (1962)	Tentative Recommended Practice for Radiographic Testing
AWS A2.0-58 (1958)	Standard Welding Symbols (American Welding Society)
AWS A3.0-61 (1961)	AWS Definitions - Welding and Cutting (American Welding Society)
SPPS 03-0010-00-C (1965)	Chemical Cleaning of Columbium, Tantalum, and Their Alloys
SPPS 03-0015-00-A (1964)	Arc Weld Groove Designs for Austenitic Stainless Steels, L-605, Columbium, and Tantalum Alloys.

3. REQUIREMENTS

3.1. Materials

3.1.1. Inert Gases. Inert gases shall be helium or argon which contain less than 1 ppm each oxygen and water vapor by volume.

3.1.2. Electrodes. Tungsten electrodes, class EWTh-2, shall conform to ASTM Designation B297-55T, "Tungsten Arc-Welding Electrodes".

3.1.3. Filler Wire. Welding filler material composition shall conform to the chemical requirements for the base metal, unless otherwise specified.

3.2. Equipment

3.2.1. Welding Machine. A direct current, arc-welding machine shall be used.

3.2.2. Chamber. The welding shall be done in an enclosed chamber that can be evacuated to less than 1×10^{-5} torr. The leak rate shall result in a pressure increase of not more than 5 microns/hour starting at 1×10^{-3} torr pressure or less. The chamber shall be equipped with glove ports and 0.030-inch thick neoprene rubber gloves that are sealed gas-tight to the ports. The gloves shall be sulphur free type and shall be qualified per paragraph 4.3.2.

3.2.3. Welding Torch. The welding torch or head shall be gas or water cooled. The water passage of a water-cooled torch or head shall be permanently sealed. Cable and hose connections shall be vacuum tight.

3.3. Cleaning and Handling

3.3.1. Components. The filler wire and parts to be joined shall be chemically cleaned in accordance with Specification SPPS 03-0010--C, "Chemical Cleaning of Columbium, Tantalum, and Their Alloys".

3.3.2. Assemblies. If intermediate processing has not maintained the cleanliness of Specifications SPPS 03-0010-00-C, the metal adjacent (nominally 4 inches on each side of the joint) to the weld shall be recleaned per SPPS 03-0010-00-C.

3.3.3. Handling. Parts, components, and assemblies shall be handled as shown on the engineering drawing or applicable specification. If no specification is denoted, parts and components shall be handled with clean, lint-free gloves.

3.3.4. Metal Vapors. Deposits of metal vapors produced by the welding process shall not be considered detrimental or subject to cleanliness requirements. These films, however, may be removed by wire brushing with refractory metal brushes.

3.4. Joint Preparations

3.4.1. Design. The edges of the parts shall be prepared for welding by machining or filing as shown on the engineering drawing, if applicable; or in accordance with Specification SPPS 03-0015-00-A, "Arc Weld Groove Designs for Austenitic Stainless Steels, L-605, Columbium, and Tantalum Alloys".

3.4.2. Fixturing. The joints to be welded shall be positioned to provide proper alignment, match of parts, and root opening. The joint edges shall not be misaligned more than 20% of the thinner section being joined or 1/16 inch, whichever is less.

3.4.2.1. Fixture Materials. The fixture components that contact the parts within 4 inches of the weld joint shall be made of molybdenum, tungsten, columbium, tantalum or their alloys. Non-refractory metals within 0.10-inch of the weld joint shall be shielded by refractory metals.

3.4.2.2. Fixture Cleanliness. The fixture shall be clean and free of surface contamination.

3.4.2.3. Tack Welds. After the parts are properly positioned, tack welds may be used to maintain alignment during welding. Tack welds shall have complete fusion and penetration to the weld joint root.

3.5. Welding Procedures

3.5.1. Qualification. Welding shall be done using equipment and materials which have been qualified per paragraph 4.3.

3.5.2. Welding Chamber. Prior to each welding cycle, the welding chamber shall be evacuated to 1×10^{-4} torr or better and heated to at least 120°F for a minimum of 4 hours. After cooling, the vacuum shall be 1×10^{-5} torr or better before filling with inert gas containing less than 1 ppm each of oxygen and water vapor by volume. Before removing the glove port cover plates, the evacuation line to the glove ports shall be closed.

3.5.3. Power Supply. Direct current, straight polarity power shall be used for welding.

3.5.4. Welding Atmosphere. Welding shall be conducted in an inert gas which does not exceed 5 ppm oxygen, 15 ppm nitrogen, and 20 ppm moisture content (10 ppm moisture content in the case of components to contain lithium). Moisture content shall be continuously monitored. Oxygen and nitrogen content shall be determined prior to welding, at 1 hour intervals thereafter, and at the completion of all welding. The calibration of the oxygen and nitrogen analysis system shall be checked before and after the welding operation by comparison with inert gas having a known impurity content. The response of the moisture analysis system shall be checked during welding by comparison with system performance recorded during chamber qualification per paragraph 4.3.3.

3.5.5. Quality Control Specimens. Prior to the first piece welded and subsequent to the last piece welded in each inert gas environment, weld bend specimens shall be prepared without filler wire addition. Each specimen shall be from 0.040 to 0.080 inch thick 0.75 inch wide (minimum), and 2 inches long (minimum), with a longitudinal weld bead about 0.25 inch wide.

NOTE: These specimens shall be retained by SPPS Quality Control for subsequent chemical analysis, should an investigation of welding contamination be desired.

3.5.6. Process. Precautions shall be taken during welding to avoid contamination of the weld metal by the tungsten electrode. The completed weldment shall remain in the inert gas environment until it has cooled below 400°F.

3.5.7. Repair. In repair welding, the weld defects shall be removed by machining or filing. Grinding is prohibited. The area shall then be rewelded, according to the requirements of this specification and reinspected.

3.6. Quality Requirements

3.6.1. General. Weld deposits shall be reasonably smooth and uniform in appearance, have complete fusion, and blend smoothly into the base metal. The welded joints shall be examined visually and by radiographic techniques, and shall be free of the following defects by these and any other method of inspection:

1. Cracks of any type or size in the weld and adjacent base metal,
2. Crater checks and cracks,
3. Surface holes,
4. Cold laps in and along the edge of the weld,
5. Overlap of weld metal on the base metal,
6. Undercutting along the edges of the weld or depression of the weld face below the adjacent base metal,
7. Weld craters,
8. Damage to the weld metal by gaseous contaminants,
9. Incorrect weld profile and size,
10. Lack of complete (100%) joint penetration in groove welds,
11. Incomplete fusion between weld metal and/or base metal,
12. Porosity in the weld metal.

3.6.2. Groove Weld Reinforcement

3.6.2.1. Weld reinforcement on the face side of the joint shall not exceed the following:

Base Metal Thickness (Inch)	Height of Reinforcement (Maximum) (Inch)
Up to 1/2	1/16
Over 1/2	3/32

3.6.2.2. Weld root reinforcement shall not be more than 25% of the tube inside diameter based on averaging of the diameters at 90° interpreted from radiographs.

3.6.3. Fillet Weld Contour. The face of a fillet weld shall be at approximately equal angles to the sections it joins, unless specifically noted otherwise on the drawing. The weld face may be slightly convex, flat, or slightly concave. Convex welds shall have a maximum convexity of 0.1 S plus 0.03 inch where S is the average length (inch) of the two legs of the fillet weld.

3.6.4. Tungsten Inclusion Limits by Radiography

3.6.4.1. The largest dimension of any single indication shall not exceed 0.010 inch in welds on material up to 0.10 inch thick and greater than 10% of the metal thickness or 0.04 inch, whichever is less, in material 0.10 inch thick and above.

3.6.4.2. The spacing between adjacent tungsten inclusions shall not be less than three times the metal thickness.

3.6.4.3. No more than three inclusions in any one inch of weld length shall be allowed.

4. QUALITY ASSURANCE PROVISIONS

4.1. Weld Inspection Procedures. All welded joints shall be inspected for conformance to the quality requirements visually and by radiographic techniques.

4.2. Radiographic Inspection

4.2.1. General Method. Radiographic procedures shall conform to ASTM E94-62T, "Tentative Recommended Practices for Radiographic Testing". Radiographic quality control procedures shall be those described in ASTM E142-64, "Controlling Quality of Radiographic Testing".

4.2.2. Penetrameters. Penetrameters shall be used for all radiographs and the penetrameter image shall be employed to determine the radiographic quality. Penetrameter design and material shall be in accordance with ASTM E142-64.

4.2.2.1. Penetrameter Placement. Penetrameters shall be employed as required by ASTM E142-64. Normally, the penetrameter is placed on the source side of the weldment. When it is not practical to place the penetrameter on the section being examined, and in the plane normal to the radiation beam, it may be positioned on a block of radiographically similar material placed as close as possible to the area being radiographed. The block shall be the same thickness as the total weld thickness, and it shall be placed so that the penetrameter is at the same distance from the film as if it were placed on the source side of the weld joint being radiographed.

4.2.2.2. Double Wall Radiography. When radiographing double-wall welds, such as joints in pipe or tubing, the thickness of the penetrameter employed shall be based upon the total thickness of material between the penetrameter and the film.

4.2.3. Weld Surface Preparation. Accessible surfaces of welds shall be prepared as necessary so that valleys between beads, weld ripples, and other surface irregularities are blended so that radiographic contrast due to surface condition cannot mask or be confused with that of any defect.

4.2.4. Pipe and Tube Joints. Welds in pipe and tube of 3 inches O.D. and smaller may be radiographed by the double wall technique and the weld zones in each wall viewed for acceptance. A minimum of two radiographs shall be taken 90° to each other. For welds in pipe or tube having an O.D. larger than 3 inches, only the weld zone closest to the film shall be viewed for acceptance. A minimum of four radiographs shall be taken 90° to each other.

4.2.5. Source to Film Distance. Minimum recommended radiation source to film distance d_o (inches) is that calculated by the following formula:

$$d_o = 2.5\ Ft$$

where

F = maximum effective radioisotope source or focal spot dimension, mm.
t = weld thickness, or pipe or tube O.D., inches

Where a gap exists between the weldment and the film holder, the minimum source to film distance should be increased by the ratio of:

$$\frac{t + gap}{t}$$

4.2.6. Radiographic Quality Level. The radiographic quality level shall be 2% (2 + 2T) as defined by ASTM E142-64.

4.2.7. Interpretation of Radiographs. The radiographs shall be examined for quality and for unacceptable weld defects described in Section 3.6. Final interpretation of radiographed welds for conformance to quality requirements is reserved by the General Electric Company.

4.2.8. Identification of Radiograph Films. A system of positive identification of the film shall be used. The following information shall appear on each radiograph and in the records accompanying each film.

1. Organization making the radiograph,
2. Date of exposure,
3. Identification of (a) the component or assembly drawings, (b) the weld serial number, and (c) the viewing direction.

4.2.9. Disposition of Radiographs. Radiographs of weldments shall be forwarded to SPPS Quality Control for filing.

4.3. Qualification of Welding Equipment and Procedure

4.3.1. Application. Before welding actual columbium or tantalum alloy parts and components, new welding equipment and inert gas chambers shall be qualified as described below. Welding equipment that has not been used for welding columbium and tantalum alloys to this specification for a period of one (1) month shall be requalified.

4.3.2. Gloves. New gloves shall be inspected for outgassing and surface bleeding by a chamber evacuation cycle to at least 1×10^{-4} torr and 120°F for 4 hours. The chamber interior and glove surfaces shall be examined for evidence of contamination following the test. During this test clean copper bars or sheet will be located in line of sight of the gloves at no more than one foot distance. Copper discoloration will be taken to result from glove outgassing, presumably sulfur, during pumpdown. This test may have to be repeated until the outgassing of the gloves has diminished or the gloves are rejected because of the outgassing or for other performance characteristics such as permeability. The gloves employed must not compromise the system qualification requirements. No gloves shall be used which show discoloration of the copper.

4.3.3. Chamber and Tooling. The weld chamber, gloves, attendant equipment, and atmosphere monitors shall be set-up as for welding with glove port covers removed after backfilling. The system shall be continuously operated for a minimum of 5 hours to observe the overall system response and atmosphere degradation rate. Weld specimens per paragraph 4.3.5. shall be prepared at the end of the 5 hours operation. Before, during and after the 5-hour operation the inert gas shall be analyzed for oxygen, nitrogen, and water vapor per paragraph 3.5.4.

4.3.4. Inert Gas Analysis System. Near the end of the 5-hour test and after welding the specimens of paragraph 4.3.5, approximately 20 ppm oxygen, 80 ppm nitrogen, and 50 ppm water vapor impurities shall be suddenly introduced into the chamber to record the monitoring system response.

4.3.5. Weld Specimen Preparation. A full penetration fusion pass shall be made on two samples of the appropriate alloy during the equipment qualification. Welding procedures shall be in accordance with the requirements of Section 3. The material thickness shall be approximately the same as the parts to be welded with a maximum thickness requirement of 1/8 inch. Each specimen shall be 2.5 inches (minimum) in length and 1.0 inch (minimum) in width. Both specimens shall be welded at the end of the five hour run, but before intentional contamination.

4.3.6. Bend Tests. One weld specimen shall be used for bend tests as described below. These tests shall generally be performed at room temperature using a bend radius equal to the base metal thickness. If these conditions are incompatible with the parent metal ductility characteristics, appropriate deviations may be made by obtaining written approval from the General Electric Company Project Engineer.

4.3.6.1. Bend Specimen. One weld specimen shall be cut 2.5 inches long (minimum) by 0.75 inch wide (minimum). The weld axis shall be centered within the specimen and parallel to the long dimension. The reinforcement on the root of the weld shall be removed before testing.

4.3.6.2. Bend Test Procedure. Bend tests shall be performed with the specimens supported on an anvil having a 75 degree vee and a 1-1/8 inch span. Three point loading shall be applied by 75 degree, radius tipped wedges. Specimens shall be placed face down and centered under the wedge with the weld axis perpendicular to the bend axis. Specimens shall be bent 90 to 105 degrees at an uniform 0.2 inch per minute ram speed. After the first bend has been made, the specimen shall be examined per 4.3.6.3., cut into halves, and each half bend tested.

4.3.6.3. Evaluation. All three bend test specimens shall be examined for cracks at a magnification of 10X and shall exhibit no evidence of cracking.

4.3.7. Chemical Analyses. Chemical analyses of the parent metal and weld metal for oxygen, nitrogen, hydrogen, and carbon shall be made from the second weld specimen. Gas analyses shall be by vacuum fusion techniques and the carbon shall be determined by the combustion method. These chemical analyses shall demonstrate that no environmental contamination occurred during welding. The following limits shall apply to the analytical results.

Carbon	±	10 ppm
Oxygen	±	30 ppm
Nitrogen	±	30 ppm
Hydrogen	±	2 ppm

4.3.8. Qualification Report. SPPS Quality Control shall maintain full records of these qualification tests.

4.4. Rejection of Weldments. Weldments and components not conforming to this specification shall be rejected.

4.5. Reports. A report shall be submitted with the finished parts and shall include the information, but not necessarily the format, of Exhbit I. This report shall be prepared for each welding cycle.

NOTE: These reports shall be retained by SPPS Quality Control, should subsequent weld evaluation be desired.

5. PREPARATION FOR DELIVERY. Not applicable.

6. DEFINITIONS

6.1. Welding Symbols. Interpretation and meaning of welding symbols on engineering drawings and specifications are those in AWS A2.0-58, "Standard Welding Symbols", unless specifically delineated otherwise in the drawings and specifications.

6.2. Welding Terminology. Welding terms in this specification are defined in AWS A3.0-61.

EXHIBIT I

PROCESS CONTROL RECORD

PROCESS CONTROL RECORD NO.

PAGE 1 OF 2

DATE	SUBJECT
1-26-66	Welding of Columbium, Tantalum and Their Alloys By the Inert Gas Tungsten Arc Process--Specification SPPS 03-0025-00-A
CONTRACT NO.	

PART NAME	DRAWING NUMBER	WELD NUMBER

A. WELDING CHAMBER: Mfgr. Model No.

(1) Vacuum Gage	Mfgr.							Model No.
(2) Readout Instrument	Mfgr.							Model No.
	Serial No.							Calibration Date
(3) Chamber Vacuum	Torr Before Bakeout							
	Torr After Bakeout (Hot)							Torr After Bakeout (Cold)
(4) Leak Rate	Minutes:	1	2	3	4	5	6	
	Vacuum (10^{-3} Torr)							Overall Microns/Hr.

B. INERT GAS: Type Supplier

(1) Inlet Analysis	H_2O PPM	O_2 PPM
(2) Inlet Gas Analysis Equipment . .	Mfgr.	Model
	Mfgr.	Model

(3) Weld Chamber Analysis: Equipment:

SCAN NO.	TIME	ANALYSIS (PPM)				CONDITION	REMARKS
		H_2O	H_2	O_2	N_2		

SP 1165

PROCESS CONTROL RECORD Continued	PROCESS CONTROL RECORD NO.	PAGE 2 OF 2

C. EQUIPMENT & PROCEDURES:

(1) Welding Equipment	Mfgr.	Model No.	
	Serial No.		
(2) Tungsten Electrodes	AWS-ASTM Class	Size	
(3) Fixtures	Material in Contact with Parts		
	Cleanliness Verification	Alignment Verification	
(4) Arc Welding Torch	Type		
	Installation Verification		
(5) Welding Power	DC Straight - Polarity Verification		
	JOINT TYPE	ARC VOLTS	INPUT AMPS
	a		
	b		
	c		
	d		
(6) Filler Wire	SIZE	MCN	CLEANING DATE
	a		
	b		
(7) Cleaning & Handling	Applicable Specification		
	Verification of Cleaning & Handling		
	Record Cleaning Process Control Record No(s).		
(8) Removal of Parts from Chamber . . .	Temperature Below 400° F by surface Pyrometer (Temp Recorded)		

D. QUALITY ASSURANCE:

	BADGE NO.	INITIALS	DATE
(1) Verification that "before & after" weld specimens are attached and this record is complete.			
(2) Visual inspection per SPPS 03 - 0025 - 00 - A			
(3) Radiographic Inspection: (a) A report similar to the attached Radiographic Test Report (SP 1164) shall be prepared and submitted to SPPS Quality Assurance along with the actual X-Ray films and this Welding Process Control Record.			
(b) If repair was required, record new Weld Process Control Number here			

	VERIFICATION OF QUALIFICATION		
(4) Equipment & Process Qualification: Last Qualification Date ____ Date of Last Weld to this Specification ____	BADGE NO.	INITIALS	DATE

SP 1165 A

GENERAL ELECTRIC
SPACE POWER & PROPULSION SECTION
CINCINNATI, OHIO 45215

RADIOGRAPHIC TEST REPORT

(A) CONTRACT NO.	(B) ASSEMBLY NAME & DRAWING NO.
(C) WELDING PROCESS CONTROL NO.	(D) LAB PERFORMING INSPECTION
(E) PROGRAM NAME	

(F) PERFORMING LAB NO.	(G) WELD NO.	(H) VIEW	(I) ORIG. REPAIR	(J) REMARKS (DISCREPANCY REPORT, ETC.)

The above radiographs have been reviewed and accepted per ____________

AUTHORIZED SIGNATURE   DATE

The above radiographs have been reviewed and accepted per ____________
except as noted in the remarks column.

AUTHORIZED SIGNATURE   DATE

SP 1164

APPENDIX B

Loop Welding Chamber Qualification Results

LOOP WELDING CHAMBER QUALIFICATION RESULTS AS PER SPECIFICATION 03-0025-00-A (Par. 4.37 Appendix A) September 21, 1968

4.3.2 Gloves - (Neo-Sol) milled neoprene, sulfur free gloves showed no discoloration of copper during bake-out of weld chamber.

4.3.3 Chamber and Tooling - The atmosphere degradation rate is given in the Process Control Record shown in Figure 23.

4.3.4 Inert Gas Analysis System - The monitoring system response was determined as follows:

A 500 cc flask containing air at room temperature was sealed shut and loaded into the welding chamber. With a calculated volume of 153 ft^3 (4.33×10^6 cm^3) it was necessary to add 4.33 cm^3 of gas for 1 ppm concentration. Corrected to STP, the 500 cc volume was equivalent to 90.2 ppm N_2 and 23.4 ppm O_2. The flask was broken at time t = 0 and the analysis for N_2 and O_2 was performed until a constant value was reached. A period of 15 min. was necessary for a complete analysis (H_2 through CO) but only the N_2 and O_2 peaks were of interest and the scan was shortened to a period of 8 minutes.

Oxygen

The system response to oxygen is shown in Figure 24. The negative values of time along the abscissa indicate the oxygen level of the chamber following the 5 hour exposure

PROCESS CONTROL RECORD

PROCESS CONTROL RECORD NO.	Corrosion Loop Chamber Qualification
PAGE 1 OF 1	

DATE	SUBJECT
9/21/68	Welding of Columbium, Tantalum, and Their Alloys by the Inert Gas Tungsten Arc Process - Specification NSP 03-0025-00-A
CONTRACT NO. NAS 3-6476	

PART NAME	DRAWING NUMBER	WELD NUMBER

A. WELDING CHAMBER: Mfgr. Vacuum Industries Inc. Model No. Special Item

(1) Vacuum Gage	Mfgr. NRC							Model No. 507
(2) Readout Instrument	Mfgr. NRC							Model No. 0710G425
	Serial No.							Calibration Date
(3) Chamber Vacuum	Torr Before Bakeout							
	Torr After Bakeout (Hot)							Torr After Bakeout (Cold) 7.7×10^{-6}
(4) Leak Rate	Minutes:	1	2	3	4	5	6	
	Vacuum (10^{-3} Torr)	038	062	083	.11	.14	.16	Overall Microns/Hr. 1.6 /hr

B. INERT GAS: Type Ultra High Purity Helium (analyzed) Supplier Matheson

(1) Inlet Analysis	H_2O 0.8 PPM	O_2 < 1.0 PPM
(2) Inlet Gas Analysis Equipment	Mfgr. NSP MARK V Gas Chromatograph	Model Special Item
	Mfgr. Panametrics Moisture Monitor	Model 1000

(3) Weld Chamber Analysis: Equipment: NSP MARK V Gas Chromatograph with Dual Detectors (Ionization Detector and Thermal Conductivity Cell)

SCAN NO.	TIME	ANALYSIS (PPM) H_2O	H_2	O_2	N_2	CONDITION	REMARKS
6	11:55	1.40	<0.50	<0.30	12.2	Back filled weld chamber	Gloves closed
7	12:05	1.60	<0.5	<0.30	12.0	" " " "	Gloves open
8	1:34	1.72	<0.50	<0.30	11.9	Weld Chamber	1 1/2 hrs
9	2:34	1.72	<0.50	0.85	11.5	" "	2 1/2 hrs
10	3:25	4.94	<0.50	1.22	12.4	" "	3 1/2 hrs
11	4:28	5.54	<0.50	1.80	12.4	" "	4 1/2 hrs
12	4:52	6.38	<0.50	1.84	12.4	" "	5 hrs
13	5:20	4.39	<0.50	1.74	12.4	" "	After welding (3) specimens

SP 1165

Figure 23. Process Control Record.

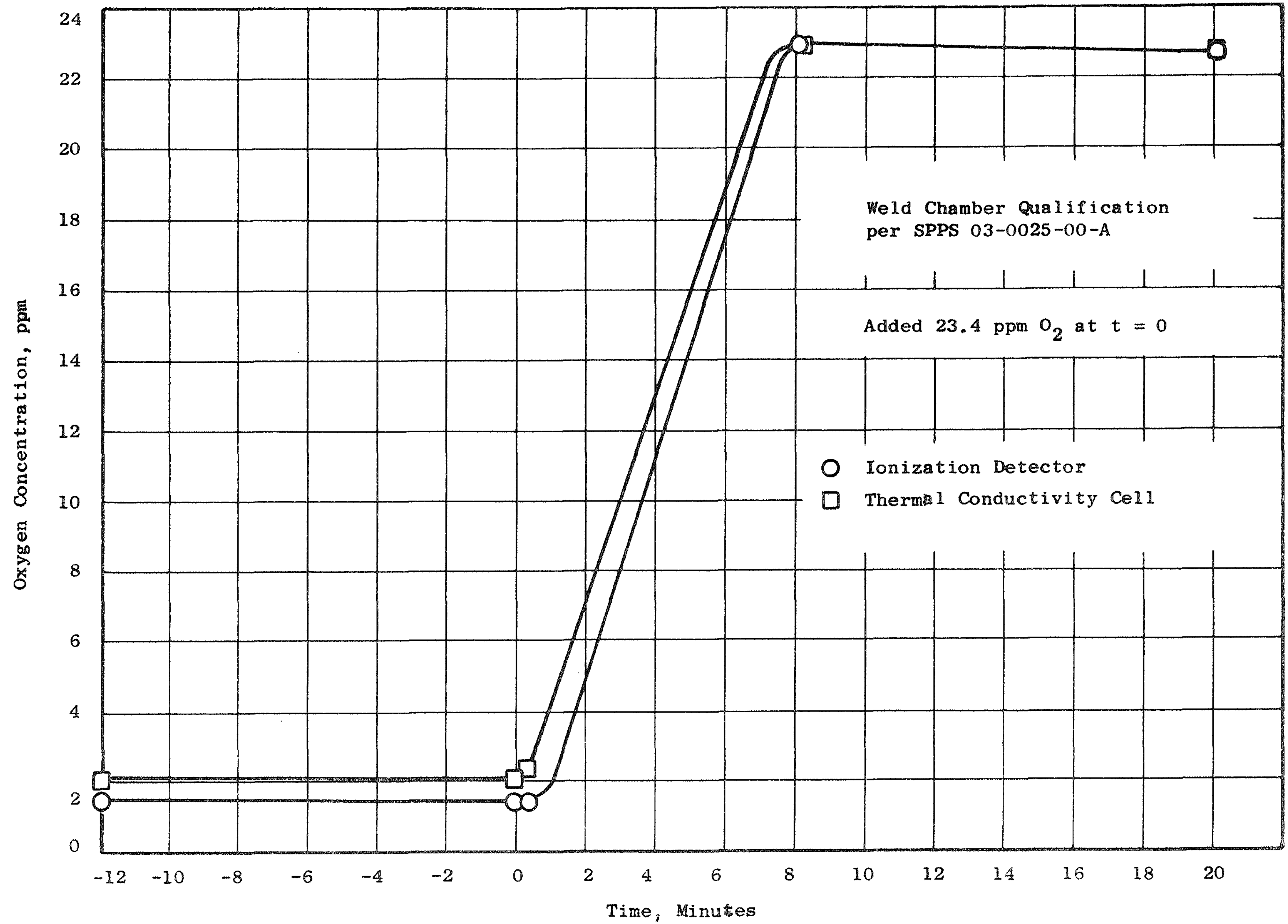

Figure 24. System Response to Oxygen.

and just prior to breaking the flask. As can be seen from the figure, both detectors were in good agreement. The average oxygen value was slightly less than 23.0 ppm as compared to the calculated 23.4 ppm introduced. The system response time was greater than 0.25 min but less than 8.0 min.

Nitrogen

The system response to nitrogen is shown in Figure 25. The ionization detector and thermal conductivity cell were in close agreement at the lower nitrogen levels but deviated at the higher nitrogen levels. Subsequent analysis of calibration mixtures with high nitrogen levels (approximately 100 ppm) indicated that the thermal conductivity cell consistently indicate lower than the ionization detector. The ionization detector indicated approximately the 90.2 ppm of nitrogen which was added. The indicated 86.3 ppm nitrogen is an error of about 3.5 percent. The system response time was greater than 0.25 min but less than 8.0 min.

Water

The system response to water vapor is shown in Figure 26. Liquid water was vaporized in approximately 30 secs. Because of the apparent low water vapor readings during the entire qualification test, the Panametrics probe #688, used during the analysis, was removed from the chamber and subsequently

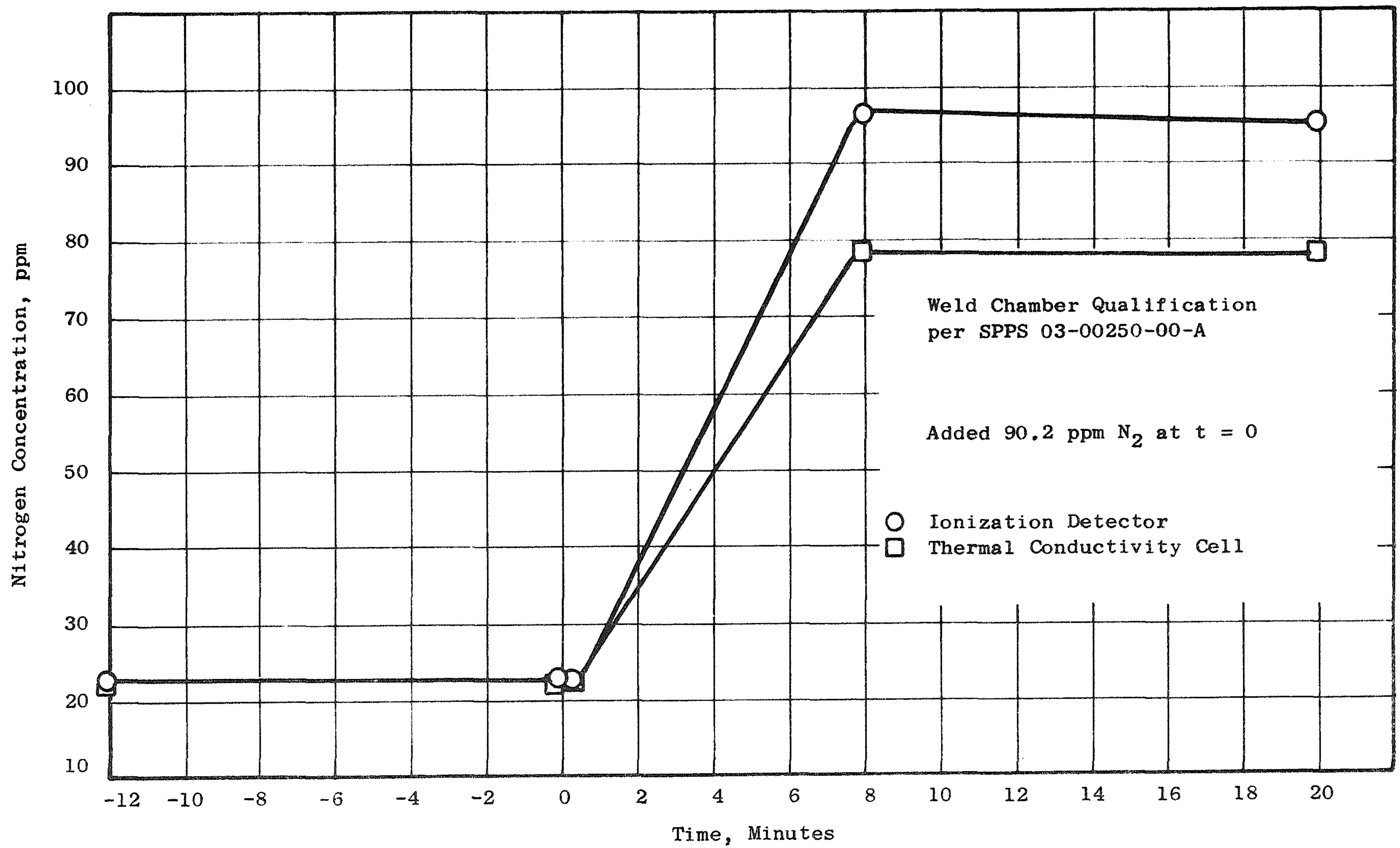

Figure 25. System Response to Nitrogen.

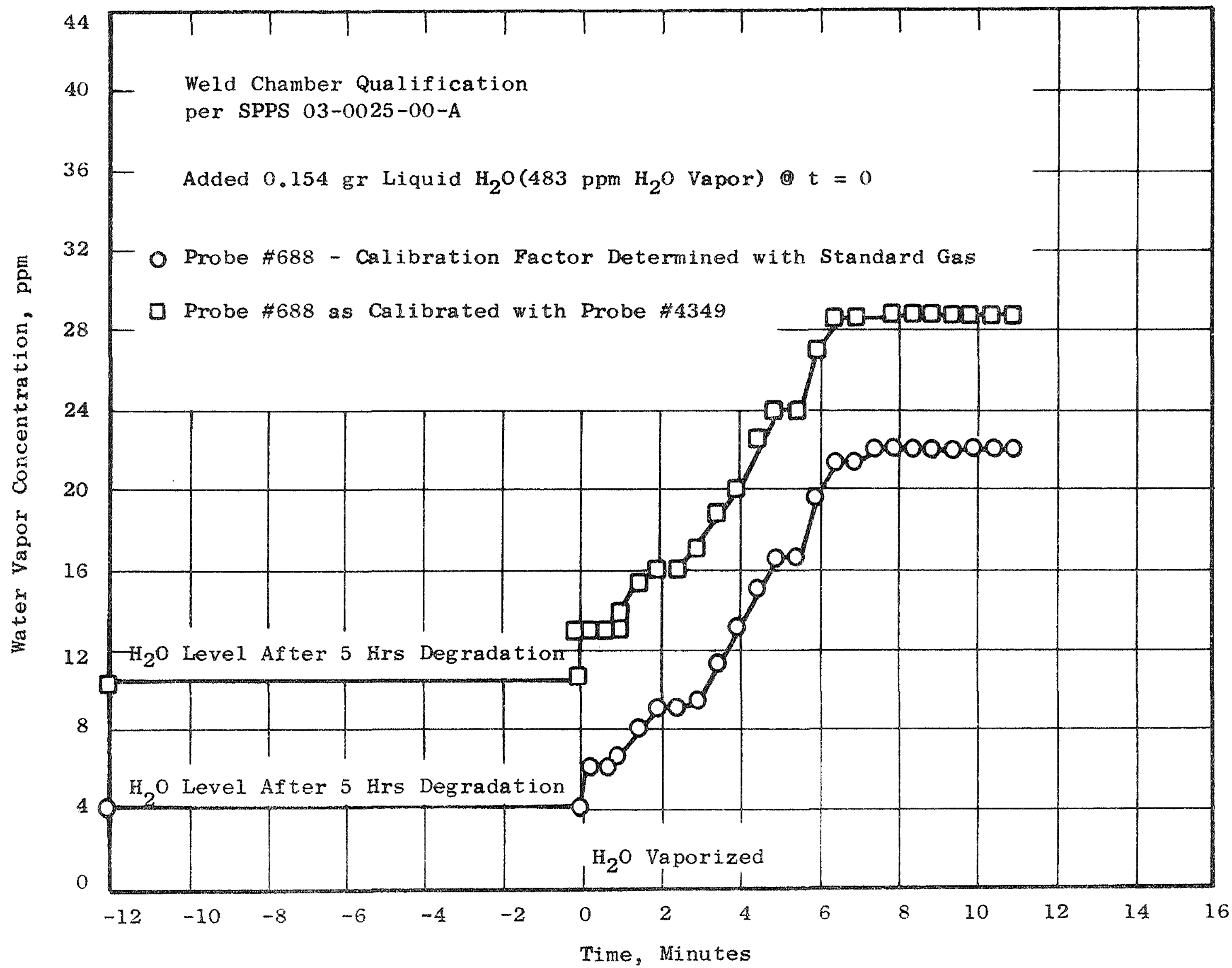

Figure 26. System Response to Water Vapor.

calibrated against a gas of known moisture concentration (48 ppm H_2O). A calibration factor (2.68 x Meter Reading) was determined and the water vapor readings were corrected. At a later date, a new probe (#4349) was run simultaneously with probe #688 in a helium atmosphere of decreasing moisture concentration. Figure 27 shows that the calibration curve, established using the new Panametrics probe #688, deviates from the true moisture reading by about 6 ppm water vapor and always reads lower than the true moisture indication. The standard gas (48 ppm water vapor) was read with probe #688 and the indicated value of moisture level was exactly that predicted by the calibration curve. Two possible explanations exist for the relatively low water vapor value (22 or 28 ppm) obtained when the water vial (48.3 ppm water vapor) was broken. It is possible that all the water was not removed from the vial and vaporized or it is possible that the chamber, being very dry with a very large amount of surface area, absorbed moisture readily. The system response time was greater than 0.5 min but less than 7.0 min.

4.3.5 Weld Specimen Preparation - Three weld specimens were prepared of T-111 sheet, 0.040 inch thick x 3 inch x 1 inch (MCN 02B-010).

4.3.6 Bend Tests - Three bends were made as required, using a 1/32-inch radius tipped wedge. All three bends showed no visual cracks after 105° bend.

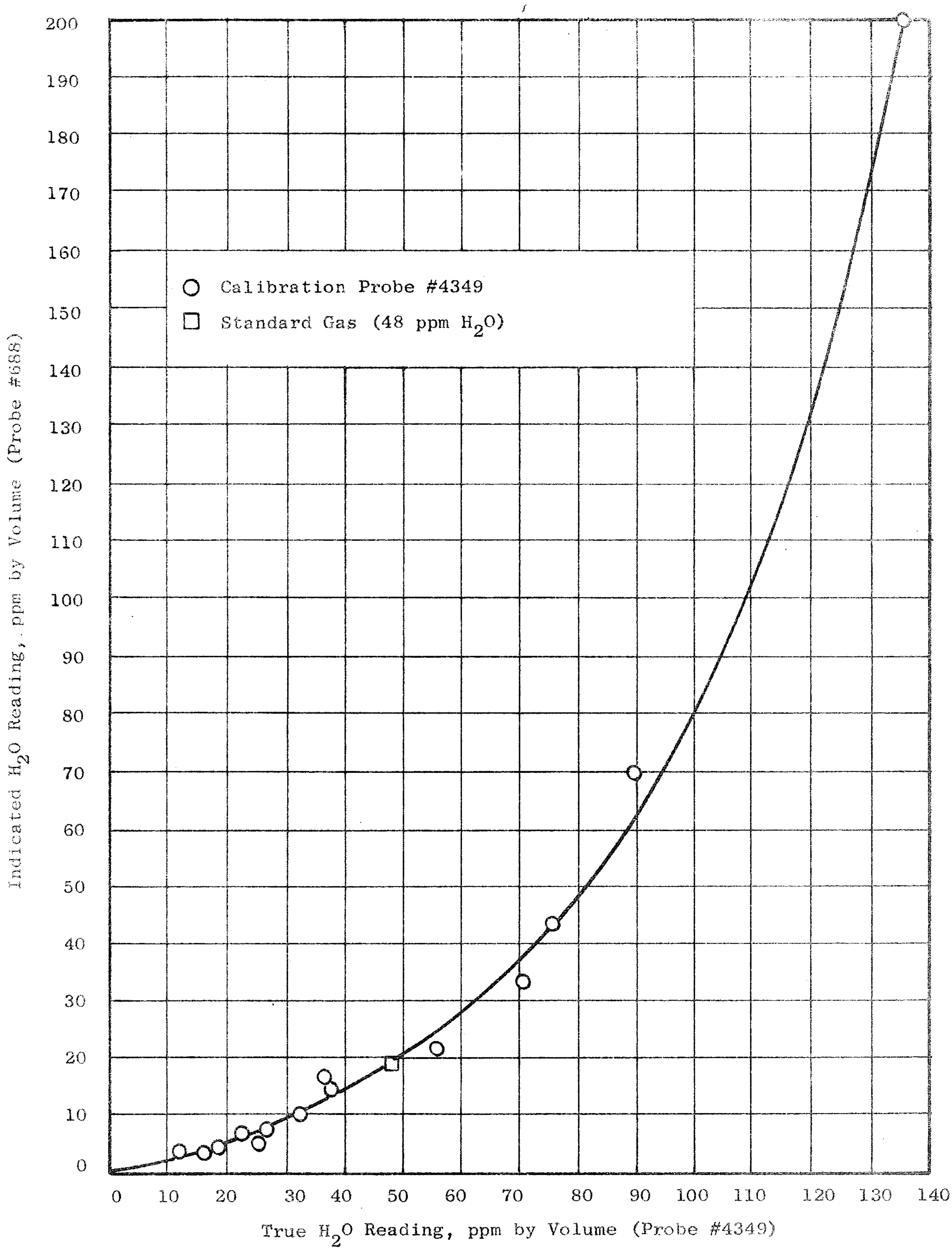

Figure 27. Calibration Curve for Panametrics Probe #688.

4.3.7 Chemical Analysis

Weld Specimen Fusion Zone:	Oxygen	77, 75 ppm
	Nitrogen	10, 11 ppm
	Hydrogen	1, 1 ppm
	Carbon	21, 32 ppm
Parent Metal (As-Received): MCN 02B-010-1	Oxygen	89 ppm
	Nitrogen	9 ppm
	Hydrogen	1 ppm
	Carbon	9, 11 ppm

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